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Development of an Accelerated Hydrogen Embrittlement Test for Manganese Phosphated Steels

G.N. Vigilante

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ABSTRACT

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A 55 hr. incremental step load test was determined to be as sensitive as the existing 200 hour sustained load test for manganese phosphate and is proposed to be available as an option for future quality assurance/quality control testing for hydrogen embrittlement.

KEYWORDS

Hydrogen embrittlement, accelerated hydrogen embrittlement test, sustained load test, SLT, incremental step load, ISL, manganese phosphate, 4340, gun steel

ABBREVIATIONS

1a.1	- Notched Tensile Specimen as referenced in ASTM F519
ASTM	- American Society for Testing and Materials
CV	- Coefficient of Variation
F&F	- Fatigue and Fracture Analysis Branch at Benét Laboratories
FDI	- Fracture Diagnostics International, LLC, Newport Beach, CA
HE	- Hydrogen Embrittlement
Hsr	- Hydrogen Susceptibility Ratio as per ASTM F2078
IG	- Intergranular
ISL	- Incremental Step Load
K_{IHE}	- Stress Intensity Threshold for Hydrogen Embrittlement
$k_{R,C}$	- One sided tolerate limit factor with a specific reliability (R) and confidence (C)
ksi	- Thousands of pounds per square inch
LRA	- Lou Raymond and Associates, Newport Beach, CA
Mn-P	- Manganese Phosphate
MVC	- Microvoid Coalescence

NFS	- ISL Notched Fracture Strength; reported as a percentage of the NTS
ISL-NFS$_P$	- Notched Fracture Strength Acceptance Criterion (via statistical analysis)
NTS	- ASTM E8 Notched Tensile Strength
PPM	- Parts Per Million
RT	- Room Temperature
QA/QC	- Quality Assurance/Quality Control
SEM	- Scanning Electron Microscope
SLT	- Sustained Load Test
s_x	- Standard deviation
WVA	- Watervliet Arsenal
$\bar{x}$	- mean

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BACKGROUND

<u>Hydrogen Embrittlement</u>

Hydrogen embrittlement (HE) is a phenomenon whereby hydrogen (in various forms) can degrade the mechanical properties of materials. In order for HE to occur, there must be at least three prerequisites which must occur simultaneously: 1) a source of hydrogen, 2) a susceptible material, and 3) a tensile stress (Figure 1). Additionally, hydrogen embrittlement tends to be most severe at or around room temperature [1]. Hydrogen can be generated/liberated as a result of many industrial processes including steel making, oil/gas drilling, chemical processing, welding, and plating, for example. The hydrogen must reach a critical concentration in order for embrittlement to occur. However, for high strength steels, this concentration need only be on the order of just a few parts per million (Figure 2) [2]. High strength steels are notoriously susceptible to HE, and slight increases in strength can profoundly affect HE susceptibility. The source of the tensile stress (applied or residual) must exceed a particular threshold (e.g. K_{IHE}) in order for HE to occur. Because of the risk of damage to components, various test methods have been established to evaluate for HE for quality assurance/quality control (QA/QC).

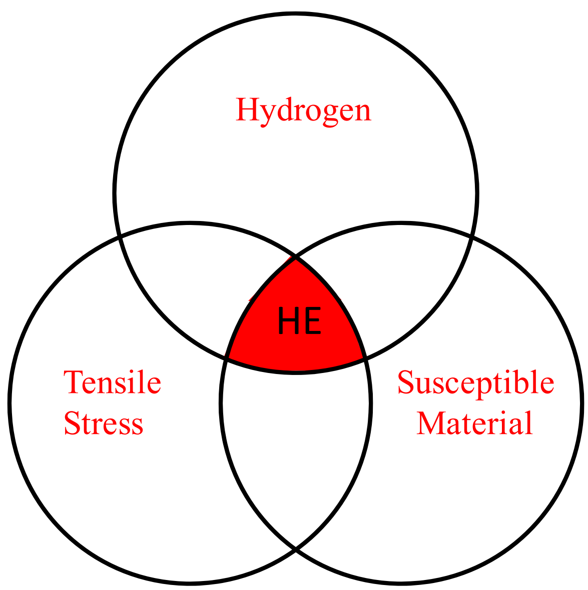


Figure 1. Venn Diagram of prerequisites for HE. The red area represents the conditions where hydrogen embrittlement will occur. Without any one variable, no hydrogen embrittlement will occur.

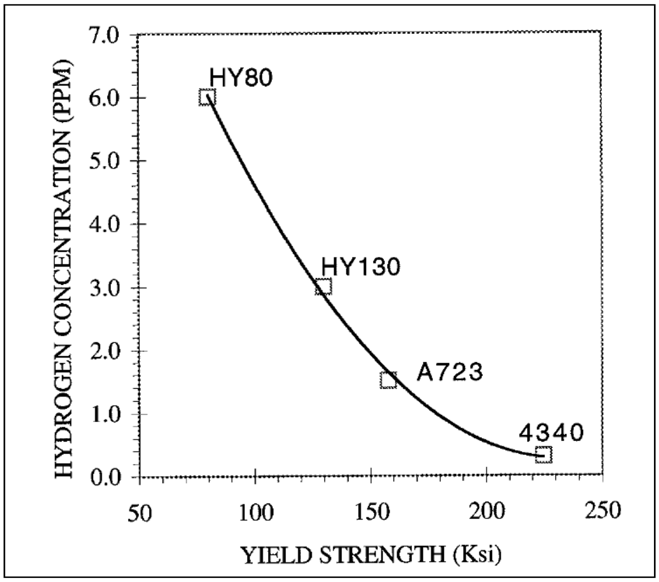


Figure 2. Effect of yield strength on the critical hydrogen concentration in some steels. From G.L. Spencer and D.J. Duquette [2].

Manganese Phosphating and Subsequent HE Testing

Manganese phosphate (Mn-P) conversion coatings are used primarily for corrosion protection. The military specification for heavy phosphate coatings (≥16 g/m^2 unless otherwise specified) is MIL-DTL-16232G.

The Mn-P process is a multi-step process that typically involves degreasing, a stress relief, abrasive blasting, brief immersion in a caustic conditioning bath, the actual phosphating process (~20 minutes at ~200 °F), water rinsing, and dipping in chromic acid (sealer). As per MIL-DTL-16232G, HE relief treatment (either 210-225 °F for 8 hrs. or 120 hrs. at RT) must be performed on any specimens/components that are ≥ HRC 39. Subsequently, QA/QC testing for HE is performed using high hardness/strength (HRC 51-53), 4340 steel, notched tensile specimens. Note: the history of MIL-DTL-16232 in relation to HE testing can be found in Appendix A. The use of notched, 4340 specimens at HRC 51-53 represents a worst case scenario due to the sharp notch (0.009 +/- 0.001 in. as per ASTM F519 Figure A1.1) and hardness/strength level (which greatly increases the HE susceptibility of the steel, Figure 2). The notched tensile specimens are loaded to 75% of their uncoated, ASTM E8 notched tensile strength (NTS) and held for 200 hrs. If two of four specimens fail this test, the coating process is considered embrittling (as per ASTM F519 which is referenced in MIL-DTL-16232G).

Incremental Step Load (ISL) Test Method

The current SLT takes too long to perform and is a qualitative (Go/No-Go) test. The requirement for a 200 hr. HE test has been "very troublesome" and has plagued metal finishing/plating industries for decades. The time between the end of the coating operation and the reporting of the HE test results can exceed two weeks (336 hrs.) when taking into consideration the 120 hr. RT delay for HE relief (if performed in lieu of the furnace bakeout), the 200 hr. SLT, any additional testing should one sample fail, and subsequent evaluation for cracking. During this time period, any additional processing of components is at risk if a HE issue is detected. Ultimately, this could lead to additional testing (and more delays), destructive examination of components, and/or scrapped components. "In a fast-paced hardware intensive program, these results can be disastrous!" [3].

A test program has recently been completed by US Army ARDEC, Benét Laboratories to develop an accelerated, incremental step load (ISL) test to evaluate for HE of Mn-P components. *The objective of this effort was to develop an accelerated (shortest duration) HE test, specifically for Mn-P, which is as sensitive as the existing 200 hr. SLT test*. Four (4) ISL machines were purchased from Fracture Diagnostics International, LLC (FDI, Newport Beach, CA) for this effort. These machines are electro-mechanically actuated and utilize displacement control. Two machines have 10,000 lbs. load cells and two (2) have 20,000 lb. load cells. Automated tensile testing (ASTM E8), sustained load testing (ASTM F519), incremental step load testing (ASTM F1624), and slow strain rate testing (ASTM G129) can easily be performed. Figure 3 shows the subject machines at Benét Laboratories. An accelerated ISL test for HE enables rapid and more

Figure 3. ISL testers at US Army, Benét Laboratories.

frequent QA/QC testing, quantitative test results (a percentage of the NTS, aka hydrogen susceptibility ratio, Hsr) , automatic generation of test reports, and data archiving. Figure 4 shows a test report from a 55 hr. ISL test in which the specimen was embrittled by hydrogen after 31 hrs. at 70% of its NTS (Hsr of 70%). Figure 5 shows another report generated from the same HE test.

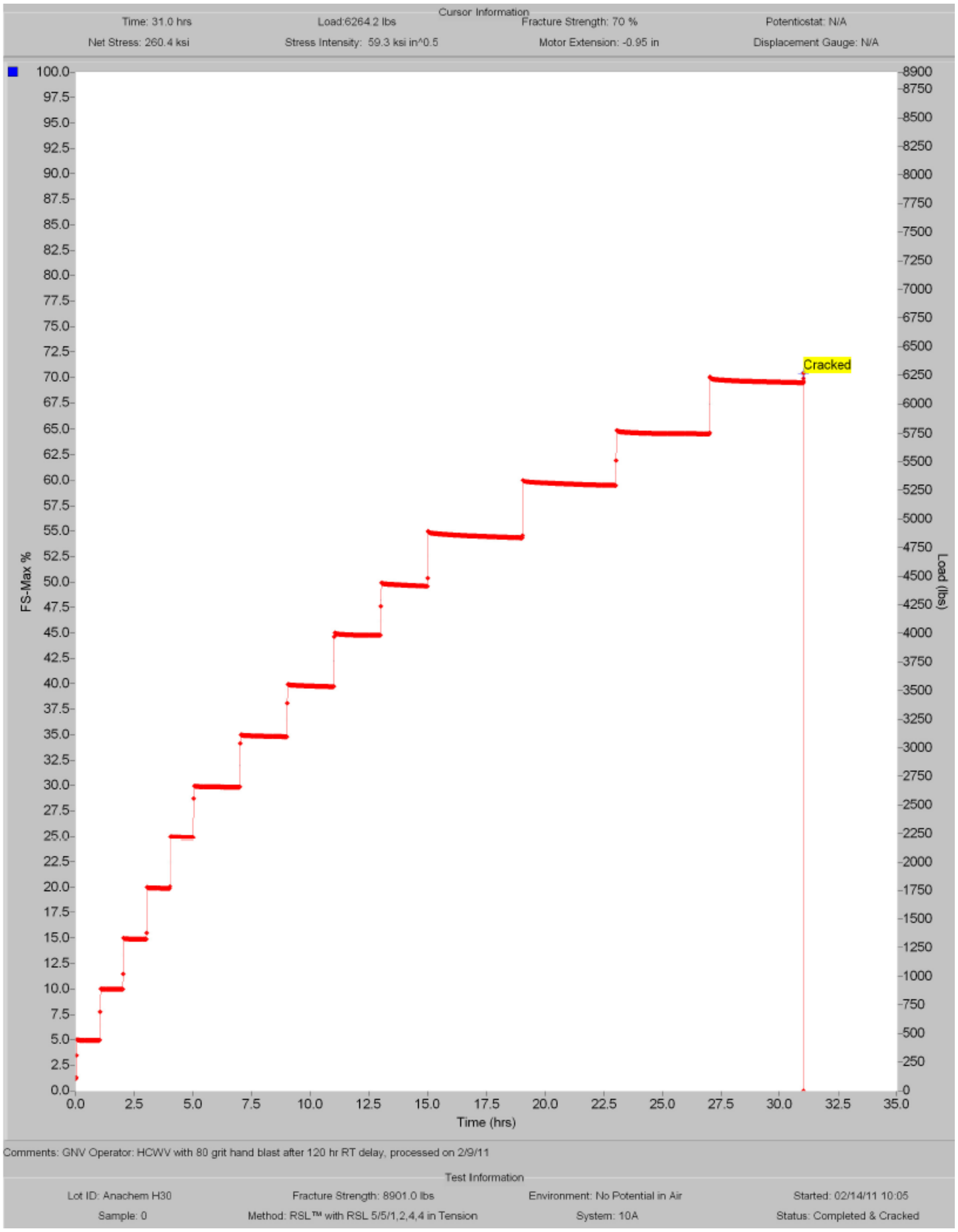


Figure 4. Example of test report from a 55 hr. ISL test (5/5/1,2,4,4).

SUMMARY OF TEST RESULTS

Circular Notched Round Bar Hydrogen Embrittlement Test

Conducted in Air

Rising Step Load, Tension

INPUT PARAMETERS

Specimen Geometry

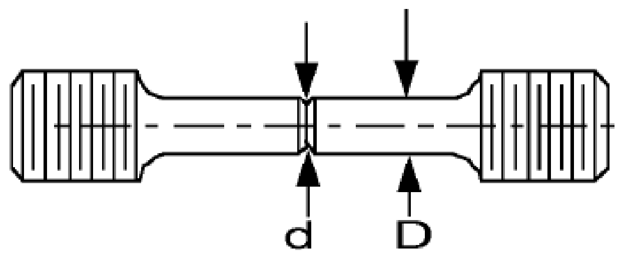
d = 0.175"

D = 0.250"

d/D = 0.700

Circular Notched Round Bar (Tension)

NRB(T)



Mechanical Properties

ASTM E8 UTS = 370.1 ksi

Fracture Load Tension = 8901.0 lbs

Computations

Applied Load, P

$$Area_{net} = \frac{\pi d^2}{4}$$

$$\text{Net Stress, } \sigma_{net} = \frac{P}{Area_{net}}$$

$$\text{Stress Intensity, } K = \sigma_{net} \frac{\sqrt{\pi d}}{2} \mathrm{f}\left(\frac{d}{D}\right)$$

$$\text{where } \mathrm{f}\left(\frac{d}{D}\right) = \frac{\sqrt{1-\left(\frac{d}{D}\right)}}{2}\left(1+0.5\left(\frac{d}{D}\right)+0.375\left(\frac{d}{D}\right)^2-0.363\left(\frac{d}{D}\right)^3+0.731\left(\frac{d}{D}\right)^4\right)$$

$$\text{Hsr} = \frac{\sigma net}{\text{UTS}_{\text{per ASTM E8}}}$$

Results

Job Title: 120 hr Delay

Material Lot = Anachem H30

Specimen # = 0

Tested by: Greg Vigilante

Test Environment = Air

Time-to-Failure = 31.0 hours

Load at Failure = 6264.2 lbs

Net Stress at Failure = 260.4 ksi

$K_{\rho IHE}$ = 59.3 ksi$\sqrt{\text{in}}$

%FS = 70.4

Hydrogen Susceptibility Ratio, Hsr = 0.70

GNV Operator: HCWV with 80 grit hand blast after 120 hr RT delay, processed on 2/9/11

Figure 5. Another test report generated from the ISL test shown in Figure 4.

The development of an accelerated, ISL test for HE was developed by Raymond et al. [4-5]. The rationale for an accelerated HE test was based on the pioneering work by A.R. Troiano [6]. Figure 6 demonstrates that notched tensile specimens of a high strength, 4340 steel loaded to 75% of its NTS can behave similarly when using a 200 hr. or 20 hr. test [3]. Of course this depends on the strength of the steel, severity of the notch, hydrogen concentration, applied stress, etc.

ISL Nomenclature

Throughout the remainder of this report, a specific notation will be used to describe the specific ISL tests evaluated. The first number represents the number of steps in the profile, the second number is the percentage of load to be applied to each step (as a percentage of the NTS), and the third number/s is the step duration in hours. For each ISL test, the summation of steps equals 100% of the NTS. For example, in a (10/5/1,2) profile, the first 10 steps are each at 5% of the NTS for a duration of 1 hr. per step, followed by 10 additional steps, each at 5% of the NTS for a duration of 2 hrs. per step. Therefore, the (10/5/1,2) ISL test is a 30 hr. test.

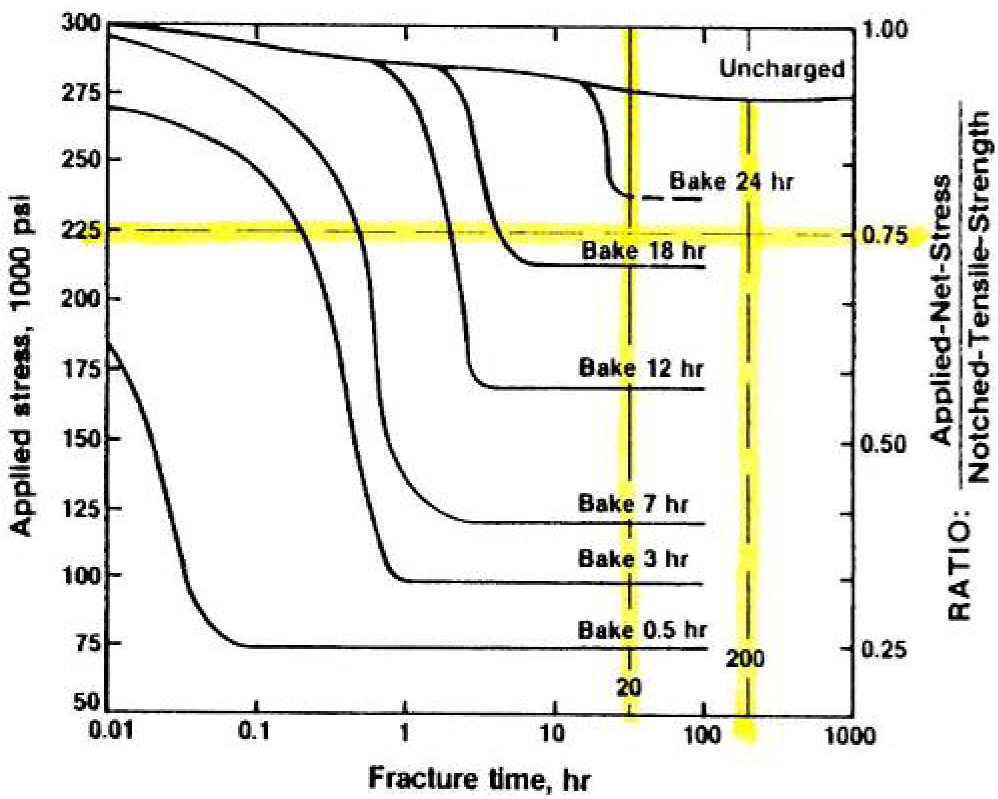


Figure 6. The effect of baking high strength 4340 notched tensile specimens at 300°F on time to failure. Note the similar performance of a 200 hr. and 20 hr. test duration at 75% NTS [4]. Used with permission.

EXPERIMENTAL PROCEDURE

Background Notched Tensile Strength (ASTM E8-NTS) Testing

For the majority of tests performed to develop and validate an ISL test for Mn-P, specimens from the same vendor (RSL Testing Systems) and lot (HT/HTP) were used. See Appendix B for the certifications for lots HT/HTP. Note: lot HTP is identical to lot HT except that the specimens were vacuum stress relieved after fabrication. Prior to performing any ISL testing, numerous ASTM E8 tensile tests were performed in order to: 1) determine if the load to failure and NTS matched that from the certification, 2) evaluate any effects from each Mn-P sub-process (e.g. stress relief, abrasive blasting, furnace bakeout, and Mn-P) on load to failure and NTS. This was an important first step in developing an ISL test since the ISL test is performed based on percentages of the NTS.

ISL Testing on Mn-P Specimens

Various ISL test profiles were evaluated in order to determine the shortest duration test that had equivalent sensitivity to the SLT. The following are examples of some profiles that were evaluated as part of this effort:

- (50% + 10/5/2) – 20 hr. test
- (15/5/1 + 5/5/2) – 25 hr. test
- (10/5/1,2) – 30 hr. test
- (10/5/1 + 5/5/2,4) – 40 hr. test
- (5/5/1,2,4,4) – 55 hr. test
- (5/5/1,2,4,8) – 75 hr. test

Figure 7 shows graphical examples of various ISL profiles vs. a 200 hr. SLT. Appendix C is a table that shows each step, duration, and cumulative duration for each of the above ISL profiles.

The majority of ISL tests were performed by the Fatigue and Fracture Analysis Branch (F&F) at Benét Labs. However, some ISL tests were also performed on identical machines by Dr. Louis Raymond, LRA, Newport Beach, CA. Comparative SLT tests were initially performed exclusively using the ISL machines, but were later also performed in conjunction with the Materials Engineering Branch of Benét Laboratories using dead-weight load machines (currently used for production QA/QC testing for HE).

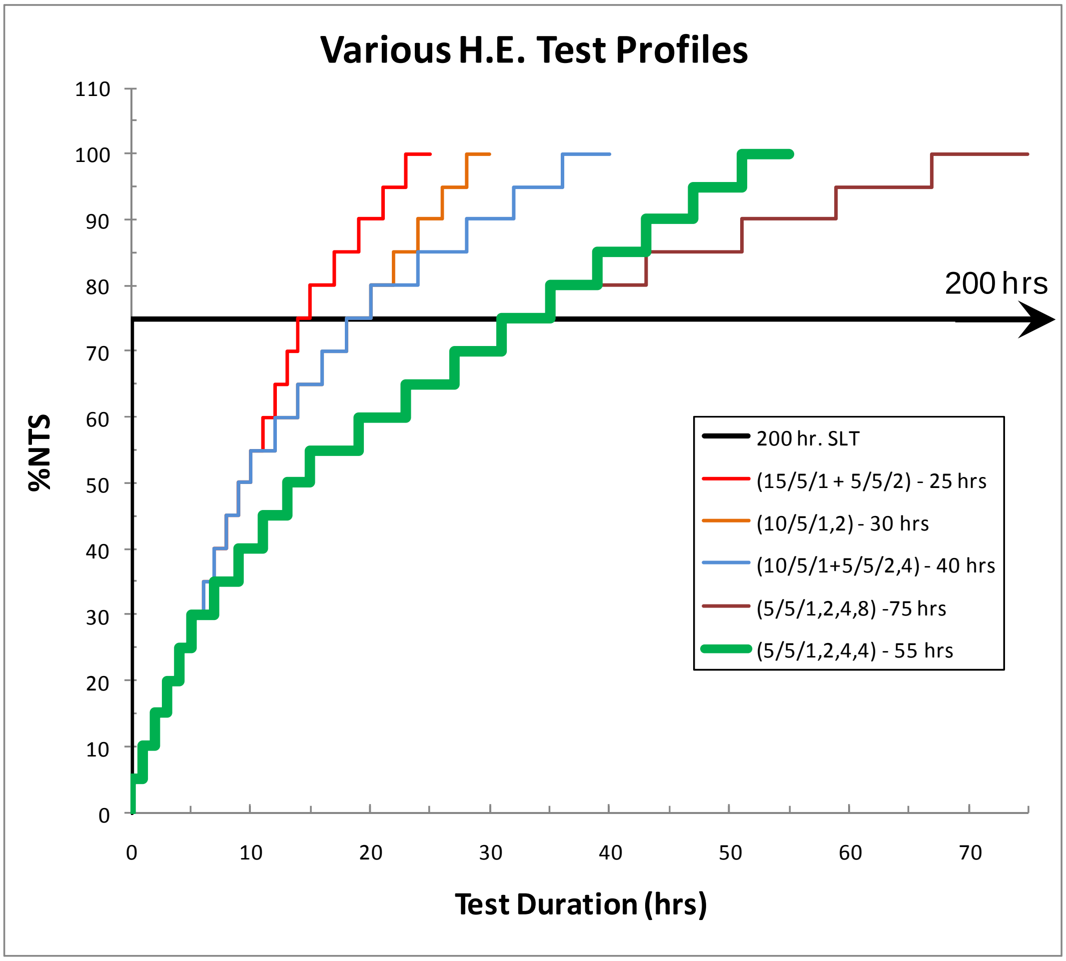


Figure 7. Examples of various ISL test profiles vs. the standard 200 hr. SLT for HE.

ASTM F1624 ("Standard Test Method for Measurement of Hydrogen Embrittlement Threshold in Steel by the Incremental Step Loading Technique") provided some general guidance for the specific ISL tests to use. In ASTM F1624, the initial ISL test to be performed on steels between HRC 45-54 is a 30 hr. ISL test (10/5/1,2). For steels between HRC 33-45, a 60 hr. initial ISL test (10/5/2,4) is recommended. In ASTM F1624, after an initial failure is observed, subsequent tests are performed at lower loads until a threshold is obtained (below which hydrogen embrittlement will not occur). This threshold can be used for design purposes, such as for plated fasteners. The various ISL tests that were performed by Benét Labs and discussed in this report bracketed the initial ISL tests recommended in ASTM F1624 as described above. Note that ASTM F1624 was not followed precisely since the objective was to develop a rapid QA/QC test method, and not to determine the threshold below which hydrogen embrittlement will not occur. A similar approach is being taken by Boeing to develop a faster QA/QC test method to evaluate for HE from cadmium (Cd) and zinc-nickel (Zn-Ni) plating baths [7].

Verification of HE

Because the hydrogen is introduced from the environment (Mn-P bath) then diffuses to regions of high triaxial stress, any embrittlement will occur locally at the notch root. This embrittlement effectively reduces the load bearing area of the specimen and results in premature failure. However, the fracture surface outside the embrittled regions remains ductile and representative of the fracture morphology of the unaffected material.

When a specimen failed prematurely (i.e. <200 hrs. for the SLT and <80-90% NTS for the ISL tests), HE was assumed. The determination of an acceptance criterion for ISL test specimens will be discussed later. In order to verify that HE occurred, numerous specimens were subsequently analyzed in the Scanning Electron Microscope (SEM). Specimens were first imaged at low magnification (~15x) to identify the presence of a contiguous shear lip around the fracture surface. For these tests, a non-contiguous shear lip is a potential sign of HE. Higher magnification imaging (1000x) was subsequently performed to identify and compare the fracture morphology at the center of the fracture surface versus the edge of the fracture surface (notch root). For quench and tempered martensitic steels, the fracture morphology changes from microvoid coalescence (MVC; ductile fracture mode) in unaffected regions to intergranular (IG; brittle fracture mode) in regions embrittled by hydrogen. Figure 8 shows a typical example of a notched tensile specimen that has been embrittled by hydrogen from a Mn-P bath. The red arrows identify multiple HE sites. The upper right image (at a HE site at the edge of the fracture surface) illustrates a brittle, IG fracture morphology compared with the bottom right image (center of specimen) which illustrates a ductile, MVC fracture morphology.

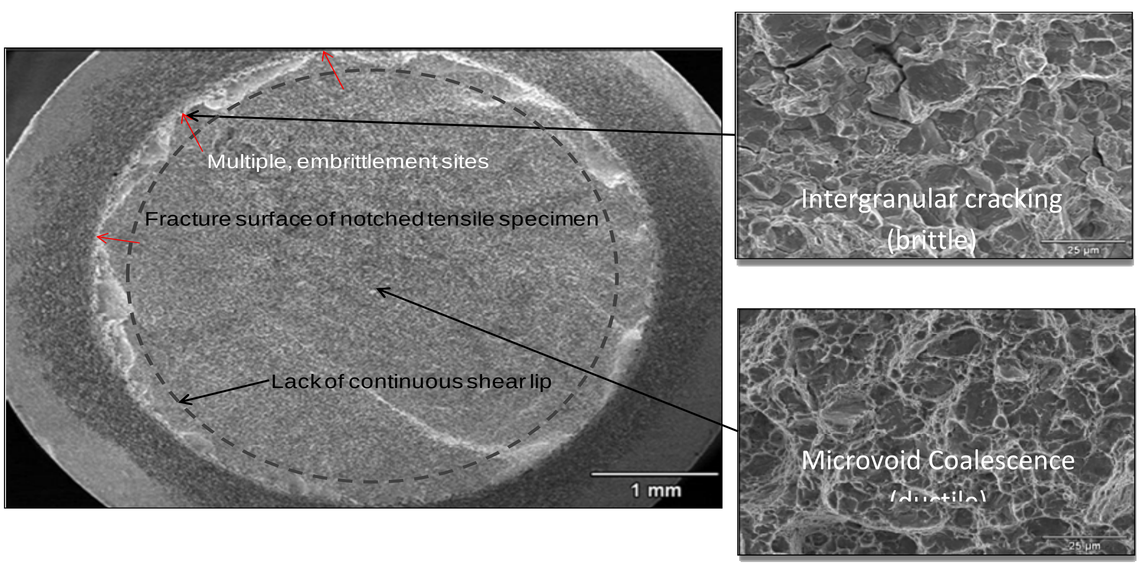


Figure 8. Example of HE in a notched tensile specimen that was Mn-P coated.

Determining the Acceptance Criterion for ISL Tests

After gaining experience with the various ISL profiles, testing was performed on uncoated notched tensile specimens (10 per ISL profile) in order to determine the load and strength at failure. Assuming a normal distribution of the test data [8, 9], a statistical analysis with a one sided tolerance limit (with a 99% reliability and a 95% confidence) was performed in order to determine the passing limit for a specific ISL test.

ISL Testing in Conjunction with Production SLT Testing

After specific ISL tests showed significant promise, testing was performed concurrently with production HE testing to gain further confidence in the ISL test profiles. Production HE testing was performed by the Materials Engineering Branch of Benét Laboratories using deadweight creep machines. Successful comparison of the ISL test method/s to production SLT tests was the final step in qualifying ISL test/s for HE testing of Mn-P components.

RESULTS AND DISCUSSION

Background Notched Tensile Strength (ASTM E8-NTS) Testing

Based on the vendor certification of the notched tensile specimens from RSL lot HT/HTP, the NTS was 373 ksi and the average load at failure was 8978 lbs. However, NTS testing performed by Benét Laboratories on a total of 38 specimens (as-received specimens and specimens that experienced each sub-process of the overarching Mn-P process) demonstrated a consistently higher average NTS and load to failure (385 ksi and 9259 lbs.) than the vendor values See Table 1 below for the average results of the Benét NTS background tests and Appendix D for the entire dataset. Because of similar results of all of the Benét background NTS tests, all results were averaged together and used going forward as the reference NTS and load to failure for

Table 1. Background ASTM E8-NTS Testing Results. Note: all specimens were taken from the same lot of material

Vendor Cert. Values

NTS (ksi)	Fracture Load (lbs)	
373	**8978**	**x-bar**
5.5	132	s_x

TEST DESCRIPTION	LOT	# Specimens Tested	NTS (ksi)	Fracture Load (lbs)	
Notched Tensile Testing on As-Received Specimens	RSL HT	18	**384**	**9233**	**x-bar**
			9.0	217	s_x

TEST DESCRIPTION	LOT	# Specimens Tested	NTS (ksi)	Fracture Load (lbs)	
Notched Tensile Testing after 375°F Stress Relief for 1 hr.	RSL HT	8	**384**	**9247**	**x-bar**
			5.9	143	s_x

TEST DESCRIPTION	LOT	# Specimens Tested	NTS (ksi)	Fracture Load (lbs)	
Notched Tensile Testing after 225°F Bakeout for 8 hrs	RSL HT	4	**383**	**9175**	**x-bar**
			11.7	306	s_x

TEST DESCRIPTION	LOT	# Specimens Tested	NTS (ksi)	Fracture Load (lbs)	
Notched Tensile Testing after Abrasive Blast w/o Mn-P	RSL HT	4	**385**	**9257**	**x-bar**
			4.3	103	s_x

TEST DESCRIPTION	LOT	# Specimens Tested	NTS (ksi)	Fracture Load (lbs)	
Notched Tensile Testing after Abrasive Blast and Mn-P	RSL HT	4	**394**	**9484**	**x-bar**
			2.7	65	s_x

Benét Results

# Specimens Tested	NTS (ksi)	Fracture Load (lbs)	
38	**385**	**9259**	**x-bar of All Benet Tested Specimens**
	8.2	202	s_x of All Benet Tested Specimens

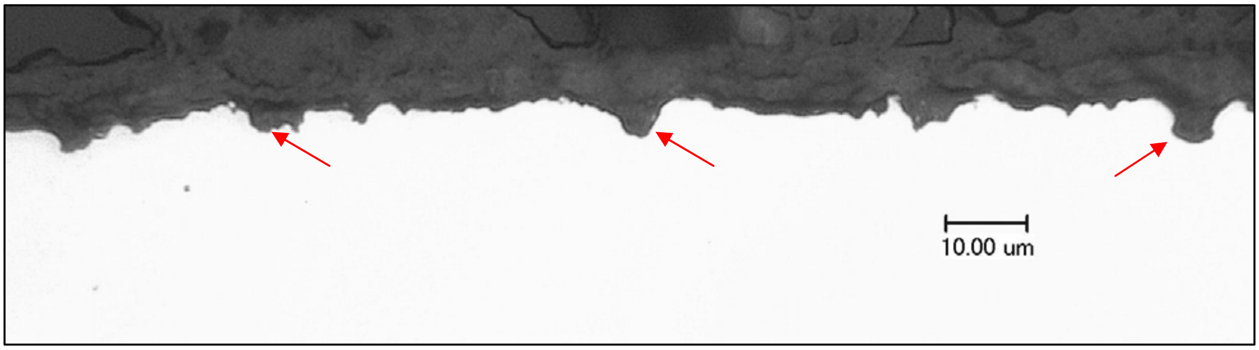


Figure 9. Photomicrograph of typical micro-pitting observed after Mn-P coating (on 4340 steel).

subsequent testing (for specimens from RSL Testing Systems Lot HT/HTP). Note that use of a higher reference NTS and load to failure is a conservative approach since the specimens will experience slightly higher loading (~3%) during subsequent ISL testing. Interestingly, the highest average NTS, load to failure, and lowest standard deviation was observed in the Mn-P coated specimens. The Mn-P process produces small pits (less than 10 microns wide/deep) on the steel substrate. These pits could act as additional stress risers and could provide a small amount of further notch strengthening. Figure 9 shows an example of the micro-pitting that occurs in the steel substrate as a result of the Mn-P process.

Determining the Acceptance Criterion for ISL Tests

A statistical analysis for a normal distribution with a one-sided tolerance limit was performed in order to determine what constitutes a "Pass". For this analysis:

$$\text{ISL-NFS}_P = \bar{x} - k_{R,C}s_x \qquad (1)$$

in which ISL-NFS_P is the notched fracture strength passing limit for a specific ISL test, $\bar{x}$ is the mean of the loads to failure, $k_{R,C}$ is a one sided tolerance factor with a reliability (R) of 99% and a confidence (C) of 95%, and s_x is the standard deviation of the loads to failure. A $k_{99,95}$ of 3.98 was used [8]. The calculated value for the ISL-NFS_P was rounded down to the nearest 5% and was subsequently used as the metric to determine if a specific ISL test passed or failed. In other words, a Mn-P specimen passes if the specimen attains ≥100% of the NFS of the bare, unplated specimen with the identical ISL loading profile. See Table 2 for the ISL-NFS_P acceptance criterion and Appendix E for the data used to determine for ISL-NFS_P for select tests of interest.

Table 2. Select ISL-NFS_P limits based on a statistical analysis for a normal distribution with a one-sided tolerance limit. See Appendix E for detailed test data.

ISL Profile	ISL-NFS_P
30 hr. test - (10/5/1,2)	≥ 90% NTS
40 hr. test - (10/5/1 + 5/5/2,4)	≥ 85% NTS
55 hr. test - (5/5/1,2,4,4)	≥ 85% NTS
75 hr. test – (5/5/1,2,4,8)	≥ 80% NTS

ISL-NFS_P testing was repeated with select ISL tests on Mn-P specimens (known to have been processed from non-embrittling baths) in order to ensure that there was no adverse affect of the Mn-P on ISL-NFS_P (e.g. due to pitting). No change in the ISL-NFS_P was observed.

The notched fracture strength, NFS, will be referred henceforth as the fracture strength of an ISL test as a percentage of the NTS. The NFS is determined from the last step that was completed plus any fraction of the next step that was completed (if applicable). For example, if the ISL specimen fractures after 75% NTS upon loading to 80% NTS, the NFS is 75%. Figure 10 shows an example of a 40 hr. ISL test (10/5/1 + 5/5/2,4) that failed after 12.8 hrs. into the test at 60% of the NTS. Because the test failed after 0.8 hrs. of a 2 hr. step at 60% NTS, the NFS is 55% + 0.8/2*5% = 57% of the NTS. Because this is < 85% NTS, this sample failed due to HE. The data from the specimen shown in Figure 10 can also be found in Appendix G.

ISL Test Results

20-30 hr. ISL tests – (50% + 10/5/2), (15/5/1 + 5/5/2), & (10/5/1,2)

In order to try to develop the shortest duration HE test possible for Mn-P, 20-30 hr ISL tests were initially evaluated. 12 head-to-head tests were performed on five (5) batches of Mn-P under room temperature delay times of <1 hr. to 72 hrs. This was considered a very aggressive test for Mn-P since the typical RT bakeout time is 120 hrs. as per MIL-DTL-16232G. 11 of the 14 SLT specimens failed these tests; however, only five of the 12 ISL specimens failed. Therefore,

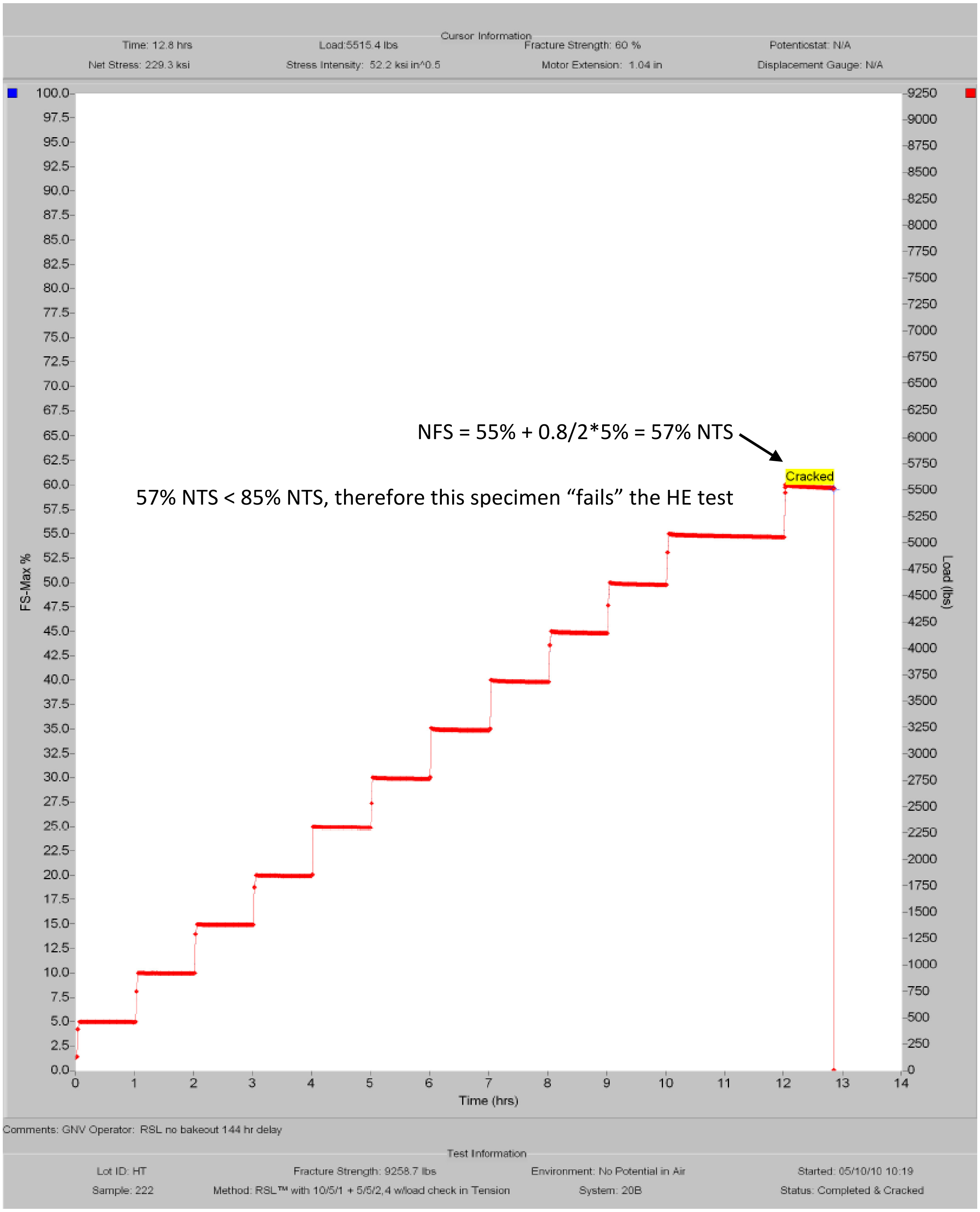


Figure 10. Example of a ISL test (10/5/1 + 5/5/2,4) that failed 0.8 hrs into a 2 hr. step at 60% NTS.

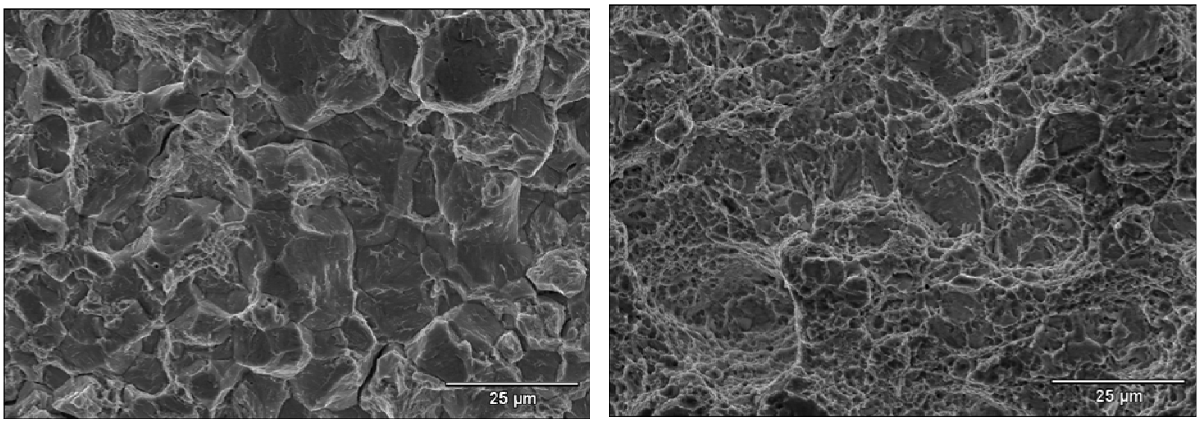


Figure 11. SEM fractography after a head-to-head HE test. IG cracking was observed on a failed SLT specimen (left image; RSL HT #35) while MVC was observed on a 30 hr ISL test that passed (right image; RSL HT 36).

these ISL test profiles were clearly not as sensitive as the SLT. For these ISL tests, there was insufficient time for hydrogen to diffuse to the root of the notch and cause embrittlement. The results for these tests can be found in Appendix F.

After HE testing, SEM fractography was performed on select specimens. Figure 11 (left) shows an example of a SLT specimen (RSL HT #35) that failed after 8 hrs. due to HE. The dominant fracture morphology was IG (brittle). Contrast this with Figure 11 (right), a SEM image of the head-to-head ISL test (30 hr.) that passed. The dominant fracture morphology was MVC (ductile).

40 hr. ISL test - (10/5/1 + 5/5/2,4)

After it was evident that the 20-30 hr. ISL tests were not as sensitive as the existing SLT, a 40 hr. ISL test was evaluated. A total of 139 specimens were tested to evaluate the 40 hr. ISL test. 33 head-to-head tests were performed on 11 batches of Mn-P under a variety of conditions ranging from testing after room temperature delays of <1 hr. to as long as 432 hrs., and testing after furnace bakeout. The majority of testing was performed on the same lot of material (RSL Lot HT/HTP) and coated by the same manufacturer (Coater A). However, some tests were also performed on two other vendor lots of material (Green Specialty Lot 38 and Anachem Lot H23) and one other coater (Coater B). Note that, as per the author's instructions, the specimens from Coater B did not receive abrasive blasting prior to Mn-P coating. Therefore, the coatings

Figure 12. Four notched tensile specimens ganged together.

produced for this investigation by Coater B are atypical and not representative of their production Mn-P process.

As can be seen in Appendix G, initial testing was performed using only one specimen per tester. However, some subsequent testing was performed by ganging up to four notched tensile specimens together in order to try to facilitate and expedite qualification testing (Figure 12).

Some of these ganged tests raised the issue of potential bending in the load train which could bias the results (results in premature failure). Consequently, flat-sided tensile specimens where strain gauged in order to determine if ganging specimens resulted in additional bending. Test results indicated bending of approximately 3-16% regardless if testing was performed on a single specimen or four (4) specimens ganged together. Additionally, testing between different ISL machines demonstrated similar results. The amount of bending that was observed is similar to what has been reported in the appendix of ASTM E1012 "Standard Practice for Verification of Test Frame and Specimen Alignment Under Tensile and Compressive Axial Force Application". Note that if any bending were to influence a test result, it would be a conservative test since the specimen would fail at a lower NFS.

Initial testing using this ISL profile was very promising. However, a few head-to-head tests had very disappointing results in which the SLT specimens were embrittled by hydrogen in a few hours while the ISL tests passed. For example, on 8/12/10, testing was performed within one hour of Mn-P. Obviously, if hydrogen embrittlement were to pose an issue, the worst case would be to test immediately after coating before the hydrogen has a change to diffuse out of the specimen. The SLT specimens failed after only a few hours into the test and exhibited clear

evidence of hydrogen embrittlement (Figure 13). However, the ISL tests passed and exhibited no clear evidence of hydrogen embrittlement (Figure 14). A similar head-to-head outcome was observed with a test performed on 7/30/10 (delay of 48 hrs. after Mn-P before testing). The SLT failed after only 12 hrs. into the test while the ISL test passed. Therefore, the conclusion is that the 40 hr. ISL test (10/5/1 + 5/5/2,4) is too fast to permit equivalent sensitivity as the 200 hr. SLT. Therefore, slower ISL tests were performed.

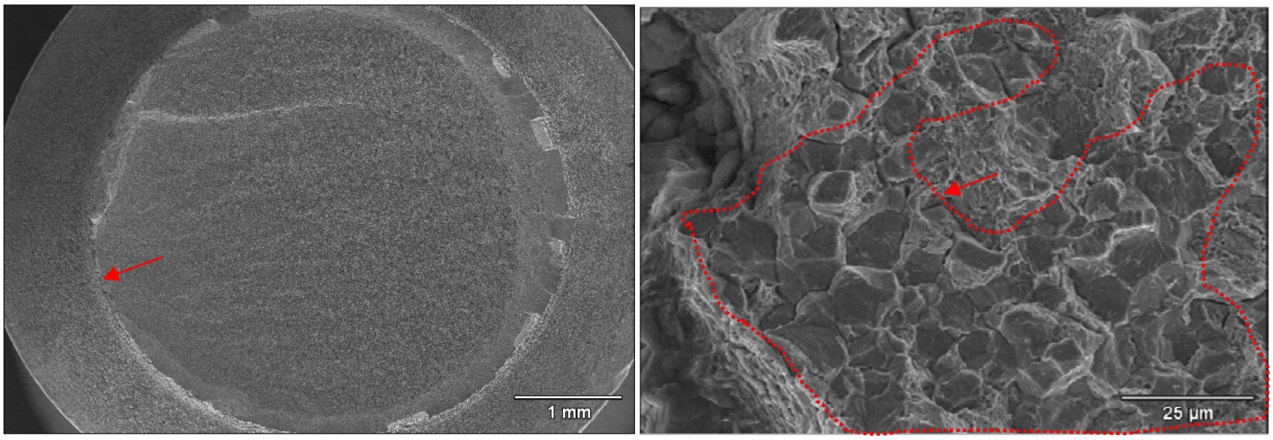


Figure 13. SLT test specimen RSL HTP #083 that *failed* after being tested within one hour of Mn-P. The left image is a low mag. SEM image showing the embrittlement site. The right image is a 1000x SEM image clearly showing intergranular cracking, a telltale for hydrogen embrittlement in quenched and tempered, high strength, low alloy steels. Note: the center of the fracture surface was ductile (MVC).

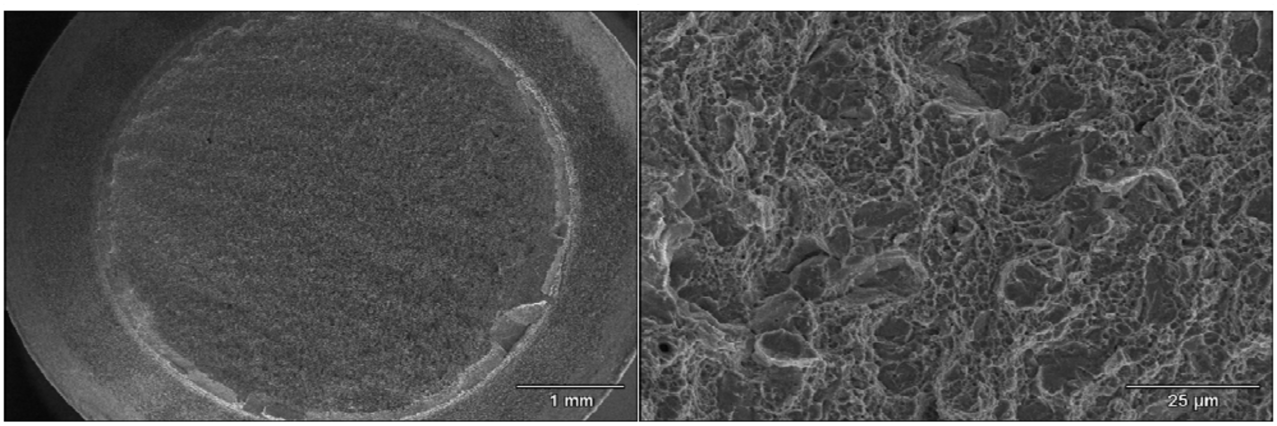


Figure 14. ISL test specimen RSL HTP #089 tested against the SLT specimen above. The ISL test *passed* after being tested within one hour of Mn-P. The left image shows a contiguous shear lip around the fracture surface and the right image shows a fracture surface at 1000x magnification in which the fracture morphology is predominantly ductile (MVC).

75 hr. ISL test - (5/5/1,2,4,8)

The 75 hr. ISL profile (5/5/1,2,4,8) was the longest duration test performed as part of this project. Note that this test is 25% longer than the 60 hr. ISL test (10/5/2,4), which is recommended in ASTM F1624 for determining the threshold of steels between HRC 33-45. Also note that the time at or above 75% NTS is 44 hrs. Therefore, it was initially thought that the longer time at these higher loads would ensure greater sensitivity. However, ISL-NFS$_P$ testing on unplated specimens demonstrated the greatest coefficient of variation (CV) of any ISL test performed (Appendix E). This higher variation resulted in a lower ISL-NFS$_P$ of ≥ 80% NTS compared to ≥ 90% NTS for the 30 hr. (10/5/1,2) and ≥ 85% NTS for the 40 hr. (10/5/1 + 5/5/2,4) ISL test profiles. The lower ISL-NFS$_P$, the decreased sensitivity, and the increased test duration makes the 75 hr. ISL test profile (5/5/1,2,4,8) inadequate to meet the objective of this project.

The reason for the higher variation is believed to be due to room temperature creep [10, 11]. Krempl states that accelerated, tertiary creep is observed when approaching the UTS of high strength steels including 4340 [11]. Tertiary creep typically occurs at high stresses and/or high temperatures in which there is an effective reduction in cross sectional area due to necking or internal void formation [12]. Others believe ISL tests fail below the NTS due to residual hydrogen present in the uncoated material [7]. The author does not share that belief as fractography was performed on numerous, uncoated, ISL specimens and no evidence of IG cracking was ever observed.

A total of 31 specimens were tested to evaluate the 75 hr. ISL test. Five (5) head-to-head tests were performed on four (4) batches of Mn-P under a variety of conditions ranging from testing after room temperature delays of 120 hrs. to 423 hrs., and testing after furnace bakeout. Of the five (5) head-to-head tests performed, only three of five tests (60%) demonstrated the same level of sensitivity as the SLT. Figures 15 and 16 show examples of a specific head-to-head test in which the SLT specimen failed while the ISL specimen passed at 80% NTS after a 432 hr. RT delay after Mn-P. See Appendix H for more information on each individual test.

55 hr. ISL test - (5/5/1,2,4,4)

Based on the previous test results, a 55 hr. ISL test (5/5/1,2,4,4) was investigated. Unlike the longer 75 hr. ISL test, ISL-NFS$_P$ testing demonstrated that the pass limit was maintained at ≥ 85% NTS for this 55 hr. ISL test (see Table II and Appendix E).

A total of 58 specimens were tested to evaluate the 55 hr. ISL test. 17 head-to-head tests were performed on nine (9) batches of Mn-P under a variety of conditions ranging from testing after RT delays of less than one hr. to greater than three months, and testing after furnace bakeout. See Appendix I for individual test results. ***Of the 17 head-to-head tests performed, all 17 demonstrated equivalent sensitivity as the 200 hr. SLT.*** Note in Appendix I that ISL specimen

RSL HTP #006 passed while SLT specimen RSL HTP #005 failed. However, this result was discounted since another SLT specimen (RSL HTP #007) from the same head-to-head test passed. See Figures 17 and 18 for a comparison of a SLT (RSL HTP #005) vs. an ISL test (RSL HTP #006). Subsequent SEM fractography revealed that IG cracking was identified on both specimens #005 and #006, even though specimen #005 (ISL test) passed (based on the statistical analysis to determine ISL-NFS_P). Because of the need for rapid interpretation of future QA/QC test results, the primary means to establish whether or not a specimen passed or failed will be based on ISL-NFS_P.

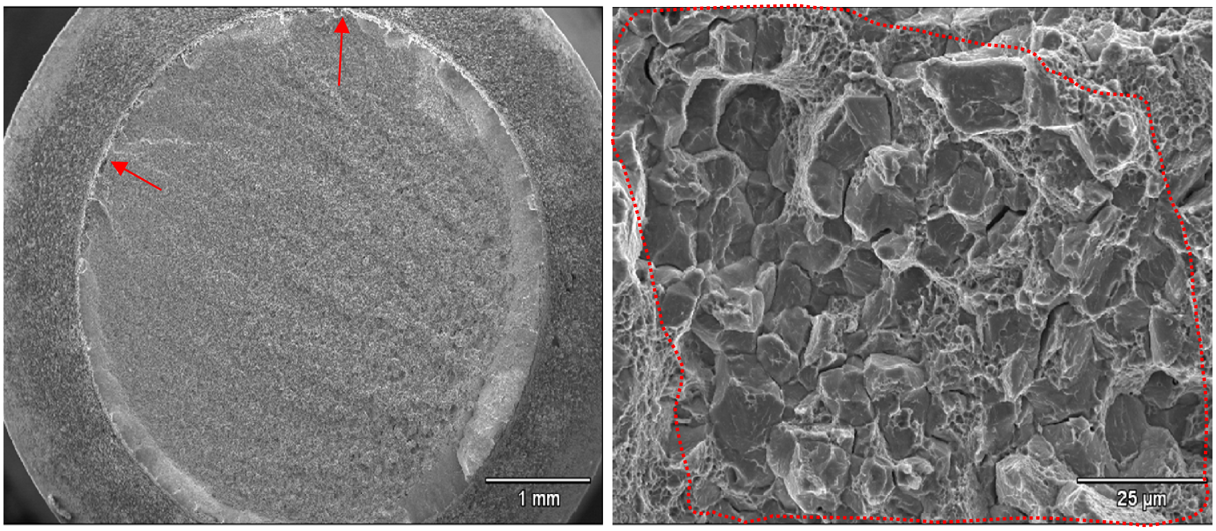


Figure 15. SLT test RSL HTP #161 that failed after a 432 hr. delay after Mn-P. The red arrows in the left image denote regions in which a non-contiguous shear lip and intergranular cracking (HE) were observed. The right image shows clear evidence of predominantly IG cracking (within the red dotted region).

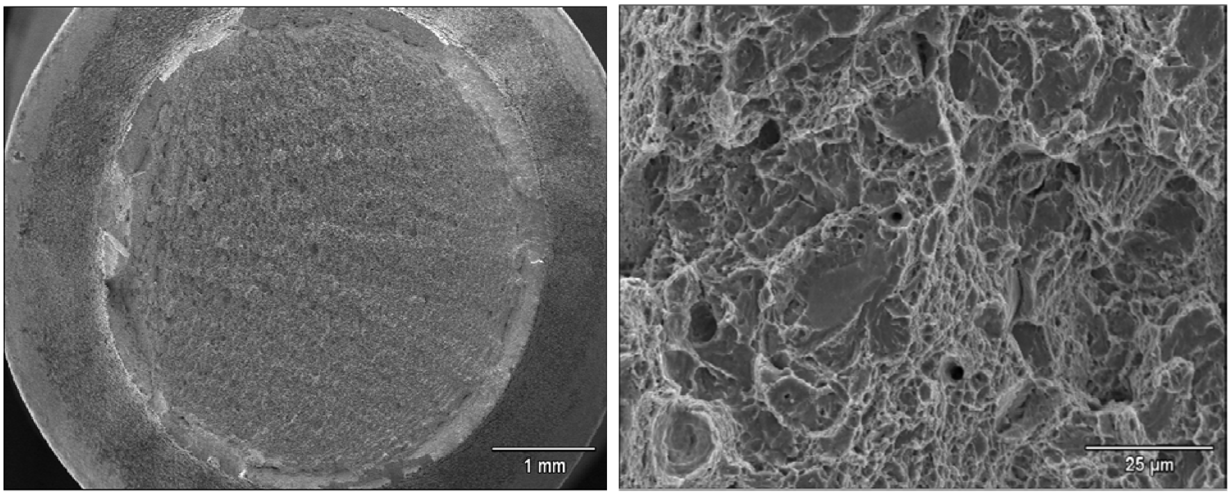


Figure 16. ISL test specimen RSL HTP #162 (75 hr. test) that *passed* after was performed after a 432 hr. delay after Mn-P. Neither the low mag. SEM inage (left) nor the 1000x mag. SEM image (right) showed any indications of HE.

Because of the observation of IG cracking in ISL specimen #006, it is likely this specimen would have failed at a higher NFS if unaffected by hydrogen (e.g. 90% NTS). However, because of the natural scatter in the ISL-NFS_P testing, the evidence of hydrogen embrittlement would have been undetected if the subsequent fractography was not performed. Because of the natural scatter of these tests, it is imperative to perform replicate testing. This will be discussed later in the ISL Test Methodology section.

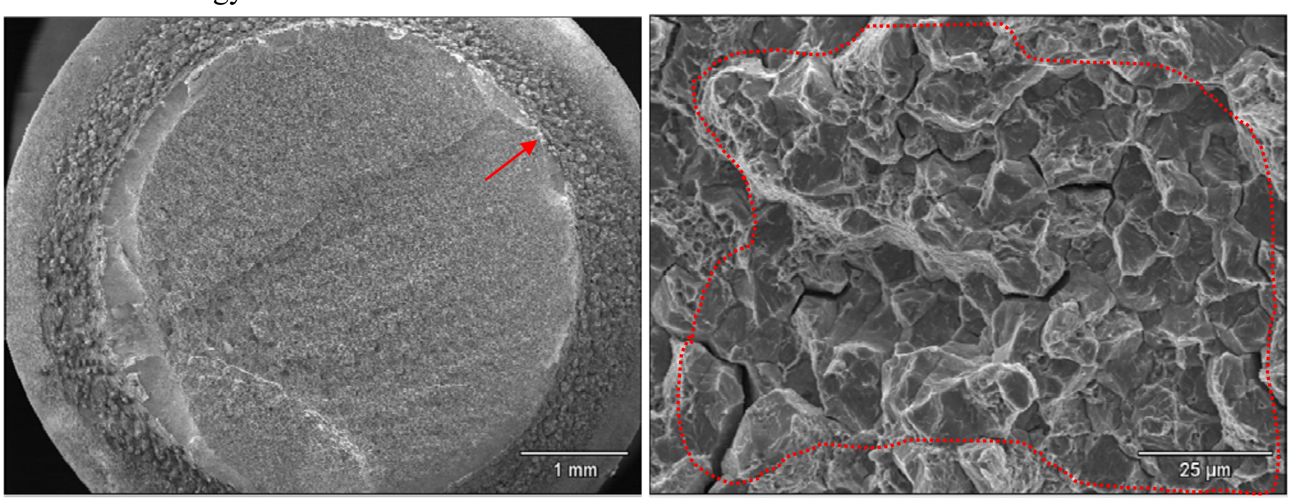


Figure 17. SLT test on specimen RSL HTP #005 that failed after a 72 hr. delay after Mn-P. The red arrow (left image) denotes the region in which a noncontiguous shear lip and intergranular cracking was observed. The right image is a 1000x SEM image which clearly shows IG cracking within the dotted red region.

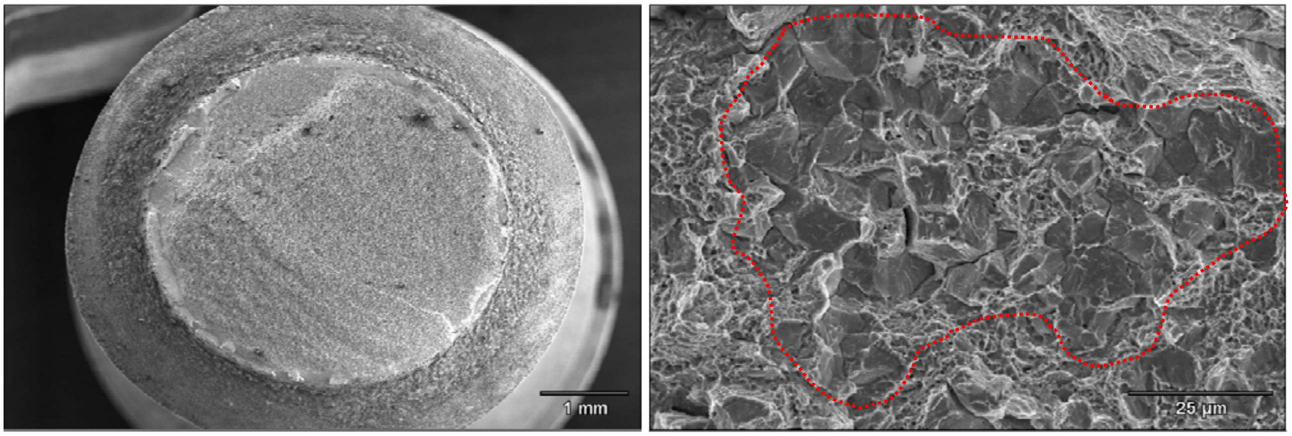


Figure 18. ISL test on specimen RSL HTP #006 that broke after a 72 hr. delay after Mn-P. This specimen was performed as a head-to-head test with specimen #005 (Figure 17). The left image is a low mag. SEM image of the fracture surface. The right image shows a region (within the red dotted area) that is clearly intergranular.

Room Temperature vs. Furnace Bakeout for HE Relief

Though not part of the objective of this effort, it should be noted that the RT delay was not equivalent to the furnace bakeout for HE relief. In all tests reported *as part of this investigation,* no furnace baked specimen failed due to HE. Numerous specimens (both SLT and ISL) from multiple batches failed after the requisite 120 hr. RT delay for HE relief. In some cases, specimens failed after as long as a 432 hr. delay after the Mn-P process (3.6x longer than the 120 hr. RT delay) while furnace baked specimens from the same batches passed. No specimens tested failed after approximately a three month RT delay, even though the particular bath was demonstrated to be embrittling based on previous failures after a 120 hr. RT delay. The reader is referred to Ref. [13] for more info. Further investigations are currently being performed at Benét Laboratories to identify the root cause/s of HE from Mn-P coatings, which do not typically cause HE [14].

ISL Test Methodology to Evaluate for HE from Mn-P baths

1) Use (2) type 1a.1, notched tensile specimens (as per ASTM F519) for each test condition of interest. Because of material strength and testing variability, it is imperative to perform replicate testing. However, based on previous testing and the low risk of H.E. in armament components due to Mn-P (see Appendix J for more info) only duplicate testing of specimens for each test condition is warranted (as opposed to four specimens as per ASTM F519).

   Example – *HE testing of both the furnace and 120 hr. RT delay is desired. Therefore, four (4) 1a.1 specimens should be Mn-P coated.*

2) Specimens should be tested for HE within one hr. of the HE relief treatment.

   Example – *For the furnace bakeout, the (2) 1a.1 specimens need to be tested within one hour of completion of the bakeout treatment (210-225°F for 8 hrs.).*

3) Load only one specimen per ISL Test Machine. Though alignment testing demonstrated no difference in bending with one specimen vs. four ganged specimens, it is prudent to only use one specimen per machine unless necessitated by test requirements. There is some concern that if one ganged specimen breaks, it could influence the subsequent behavior (in a retest) of the remaining specimens [7].

4) After installing a test specimen, ensure that there is adequate slack in the load train so that the end couplers are free to self-align during application of the load.

   Example – *The bolt extending from each coupler should be free to rotate 360° after the specimen has been installed.*

5) Establish a test file with a unique identifier for data archiving purposes.

   Example – "*110228_HCWV Furnace Bake_10A*". Use of the date first in YYMMDD format facilitates records management and future data searches. The date is followed by a brief descriptor (the coater and the condition evaluated) and the identification of which ISL test machine used.

6) Select the test parameters.

   Example – *Enter specific ID, Lot #, Batch Description, specimen type (1a.1 notched tensile specimen), test profile [i.e. (5/5/1,2,4,4)], test operator, and any other pertinent comments.*

7) Zero out the load cell. More information is found in the test manual.

8) Pre-load each specimen to approximately 100 lbs. More information is found in the test manual.

9) Select "Run" to begin the test. More information is found in the test manual.

10) After test completion, determine if the test "Passed" or "Failed".

   <u>Example 1 (Both Specimens Pass).</u> Two ISL specimens are tested (5/5/1,2,4,4) after a furnace bakeout (210-225°F for 8 hrs.). The first specimen breaks after 47 hrs. upon loading to 95% NTS. The second specimen breaks after 45 hours after two hours at 90% NTS. The first specimen has a NFS of 90% NTS and the second specimen has a NFS of 85% + 2/4*5% = 88% NFS. Both specimens exceed the ISL-NFS_P limit, and therefore pass. No further actions are necessary.

   <u>Example 2 (One Specimen Passes & One Specimen Fails).</u> Two ISL specimens are tested (5/5/1,2,4,4) after a 120 hr. RT delay. The first specimen breaks after 43 hrs. upon loading to 90% NTS. The second specimen breaks after 42 hrs. (3 hrs. at 85% NTS). The first specimen has a NFS of 85% NTS and passes. The second specimen has a NFS of 80% + 3/4*(5%) = 84% NTS. This specimen is below the NFS_P limit and fails; therefore, the bath is considered suspect, but passes. At the discretion of the production facility or the engineering authority, an immediate retest may be performed, but is not mandatory. A low magnification loupe *may* be used to inspect the specimen to try to determine if HE occurred. As previously mentioned, a noncontiguous shear lip may be an indicator for HE. Higher magnification fractography in the SEM or another instrument *may* be performed but is not necessary.

   <u>Example 3 (Both Specimens Fail).</u> Two ISL specimens are tested (5/5/1,2,4,4) after a 120 hr. RT delay. The first specimen breaks after 37 hrs. (2 hrs. at 80% NTS). The second

specimen breaks after 39 hrs. upon loading to 85% NTS. The first specimen has a NFS of 75% + 2/4*5% = 78% NTS. The second specimen has a NFS of 80% NFS. Both specimens broke below the ISL-NFS_P limit of ≥ 85% NTS. Since both specimens broke at <85% NFS, the Mn-P process is considered embrittling, pending subsequent inspection of the specimens. A low magnification loupe or other instrument must be used to evaluate the fracture surface appearance. As previously mentioned, a noncontiguous shear lip may be an indicator for HE. Higher magnification fractography in the SEM or another instrument must be performed to verify that HE occurred (IG cracking) and was not due to materials or fabrication defects (e.g. inclusions, abusive grinding, etc.). If it is conclusively determined that HE occurred, then the Mn-P process is considered embrittling and corrective actions including immediate retesting must be performed.

SUMMARY

- Hydrogen embrittlement (HE) can occur in steels as a result of numerous industrial processes, including metal plating and finishing. HE, even if present in ppm levels, can degrade steel mechanical properties and result in premature and catastrophic failure.
- The current QA/QC test to evaluate for HE in manganese phosphate (Mn-P) coated steels is a Sustained Load Test (SLT) in which notched tensile specimens are loaded at 75% of their notched tensile strength for 200 hrs.
- Various incremental step load (ISL) test profiles were evaluated against the 200 hr. Sustained Load Test (SLT) in order to try to develop and qualify an accelerated HE test specifically for Mn-P coated steels.
- The majority of tests was performed by one coater and used notched tensile specimens from the same lot of material.
- Replicate ISL testing and a statistical analysis (one sided tolerance limit of a normal distribution) was performed on uncoated notched tensile specimens in order to determine the passing limit for various ISL tests with a 95% confidence and a 99% reliability. The passing limit for 30 hr., 40 and 55 hr., and 75 hr. ISL test profiles were ISL-notched fracture strengths ≥ 90%, 85%, and 80% of the original, ASTM E8-notched tensile strength, respectively.
- Subsequent "head-to-head" ISL vs. SLT tests were performed on numerous Mn-P coated specimens. If ISL specimens failed below their passing limit or SLT specimens failed before 200 hrs., HE was assumed and later verified through fractographic analysis in the scanning electron microscope. Indicators of HE include a noncontiguous shear lip around the fracture surface and localized intergranular cracking surrounded by ductile fracture.
- 20-40 hr. ISL tests were too short to allow a sufficient concentration of hydrogen to collect at the notch root and cause HE. Several of these shorter duration ISL tests "passed" while the SLT tests failed.
- A 75 hr. ISL test profile was also not as sensitive as the SLT. A major issue with the 75 hr. ISL test was a lower passing limit believed to be due to tertiary creep at high loads.

CONCLUSION

A 55 hr. ISL test was found to be as sensitive as the SLT after evaluating a total of 58 specimens in 17 head-to-head tests on nine (9) different batches of Mn-P. These tests are approximately 4x faster than the existing SLT and are recommended for use as an option for use in future QA/QC testing for HE in Mn-P coated steels. A testing methodology for the 55 hr. ISL test is provided for guidance.

ACKNOWLEDGEMENTS

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APPENDIX A

History of MIL-DTL-16232 in relation to addressing hydrogen embrittlement

1951: MIL-DTL-16232 (spec. for heavy manganese phosphate) established.
For Alloy Steels >HRC 40: Furnace bakeout at 210-220°F for 1hr. No RT "bakeout" specified.

1958: For steels ≥ HRC 40. Furnace bakeout at 210-225°F for 1 hr. Option of a RT "bakeout" was introduced (>64 hrs).

1964: For alloy steels ≥ HRC 39 and carbon steels ≥ HRC 47. Furnace bakeout of 210°F - 225°F for 8 hrs. Doubled RT "bakeout" time to 128 hrs.

1966: Use of regular/notched tensile specimens tested at 75% of their YS/NTS for 200 hrs. To evaluate for HE.

1974: Decrease in RT "bakeout" from 128 to 120 hrs.

1978: Perform HE testing on all parts for which HE relief is required (now *ALL* steels ≥ HRC 39) and on parts which are loaded to >25% of their YS during service. Option of furnace bakeout at 210-225°F for 8 hrs. or 300-350°F for 4 hours (when approved) or RT "bakeout" for 120 hrs. Notched tensile testing is performed to evaluate for HE.

1992: Deleted the option of HE relief treatment at 300-350°F for 4 hrs. Q.C. testing for H.E. as per ASTM F519.

2000: Latest Revision (G).

APPENDIX B

Certification Sheet for Notched Tensile Specimens from Lot HT

FRACTURE DIAGNOSTICS INT'L, LLC

CERTIFIED *RSL*™ TEST SPECIMEN

CERTIFICATION

FDI

ASTM F 519 Type 1a.1, CIRCUMFERENTIALLY NOTCHED ROUND TENSILE BARS

MATERIAL: AISI 4340 PER MIL-S-5000E

HEAT TREAT CONDITION: HEAT TREAT TO SAE AMS 2759/2 DOUBLE TEMPER @425 F TO HRC 51-53

CONFIGURATION: IDENTIFICATION: CONFORMING TO ASTM F519—TYPE 1a.1 AS PER DRAWING FDI/RSL D01-REV. 9-96 (AVAILABLE ON REQUEST). THIS DRAWING DESCRIBES A SAMPLE THAT IS 2.2" LONG, .250' ROUIND IN THE TEST CROSS-SECTION, WITH A NOTCH THAT HAS A 60° INCLUDED ANGLE AND A ROOT RADIUS OF .009"

THIS CERTIFICATION ACCOMPANIES A LOT OF SPECIMENS WITH THE FOLLOWING IDENTIFICATION:

DATE SHIPPED: 2-25-09

CUSTOMER: US Army ARDEC

NO. OF SPECIMENS: 100

ADDRESS: Richard Resue
US Army ARDEC
Bldg. 115 1 Buffington St,.
Watervliet, NY 12189

LOT IDENTIFICATION: HT
SERIAL NO.'S 001—100

FRACTURE STRENGTH 373 KSI

8978 LBS

SENSITIVITY Pass per ASTM F 519 Sec. 7.3
3-Samples @ -1.2V<50% (40%, 40%, 40%)
3-Samples in air ≥ 75% (87.4%,92.3%, 88.1%)

ROCKWELL HARDNESS 52.8 HRC

CONTACT: Greg Vigilante

P.O. NUMBER: Verbal

P.O. DATE: Feb 23, 2009

CHEMISTRY:	SPECIFIED	THIS LOT
Cr	0.70 - 0.90	0.81
Cu	0.35 Max	0.16
Mn	0.65 - 0.85	0.75
Mo	0.20 - 0.30	0.21
Ni	1.65 - 2.00	1.68
P	0.025 Max	0.008
Si	0.15 - 0.35	0.25
C	0.38 - .043	0.41
S	0.025 Max	0.003
Fe	Balance	

THE ABOVE INFORMATION IS HEREBY CERTIFIED TO BE TRUE AND ACCURATE.

FRACTURE DIAGNOSTICS INT'L, LLC

FRACTURE DIAGNOSTICS INT'L, LLC PO BOX 10239 Newport Beach, CA 92658
Phone: 877 572 8541 Fax: 949 474 9807 Email: sales@fracturediagnostics.net

APPENDIX B, Con't

Certification Sheet for Notched Tensile Specimens from Lot HTP

RSL™ Testing Systems

CERTIFIED TEST SPECIMEN

CERTIFICATION

MATERIAL	AISI 4340 PER MIL-S-5000E
HEAT TREAT CONDITION	QUENCHED & TEMPERED PER MIL-H-6875H to HRC 51-53
CONFIGURATION: NOTCHED ROUND BAR	CONFORMIMG TO ASTM F519 – TYE 1a.1 AS PER DRAWING FDI/RSL D01-REV. 9-96 (AVAILABLE UPON REQUEST). THIS DRAWING DESCRIBES A SAMPLE THAT IS 2.2" LONG, 0.250" ROUND IN THE TEST CROSS-SECTION, WITH A NOTCH THAT HAS A 60° INCLUDED ANGLE AND A ROOT RADIUS OF 0.009" PROCESSED AS PREMIUM GRADE
IDENTIFICATION	EACH SPECIMEN SHALL HAVE THE LOT NUMBER AND SERIAL NUMBER ENGRAVED ON ONE END.

THIS CERTIFICATION ACCOMPANIES A LOT OF SPECIMENS WITH THE FOLLOWING IDENTIFICATION:

CONTACT	Greg Vigilante
ADDRESS	US Army Ardec Building 115 1 Buffington St Watervliet, NY 12189
P.O. NUMBER	PORF: 17519
P.O. DATE	March 3, 2010

DATE SHIPPED	May 4, 2010
NO. OF SPECIMENS	80
LOT IDENTIFICATION	HTP
SERIAL NO.'S	001 - 080
FRACTURE STRENGTH	373.3 ksi / 8978 lbs
ROCKWELL HARDNESS	52.8 HRC
SENSITIVITY	Pass per ASTM F 519 Sec. 7.3 3-Samples @ -1.2V<50% (40.0%, 40.0%, 40.0%) 3-Samples in air ≥ 75% (87.4%,92.3%,88.1%)

CHEMISTRY	SPECIFIED	THIS LOT
Cr	0.70 – 0.90	0.81
Cu	0.35 Max	0.16
Mn	0.65 – 0.85	0.75
Mo	0.20 – 0.30	0.21
Ni	1.65 – 2.00	1.68
P	0.025 Max	0.008
Si	0.15 – 0.35	0.25
C	0.38 – 0.43	0.41
S	0.025 Max	0.003
Fe	Balance	

THE ABOVE INFORMATION IS HEREBY CERTIFIED TO BE TRUE AND ACCURATE.

Tested & Certified by FRACTURE DIAGNOSTICS, INT'L, LLC
Newport Beach, CA 92660

RSL™ **Testing Systems, 168 Posey Rd. Natchitoches, LA 71457**
PHONE 318 354 0270 FAX 318 354 0271 Email: sales@rsltestingsystems.com

APPENDIX C

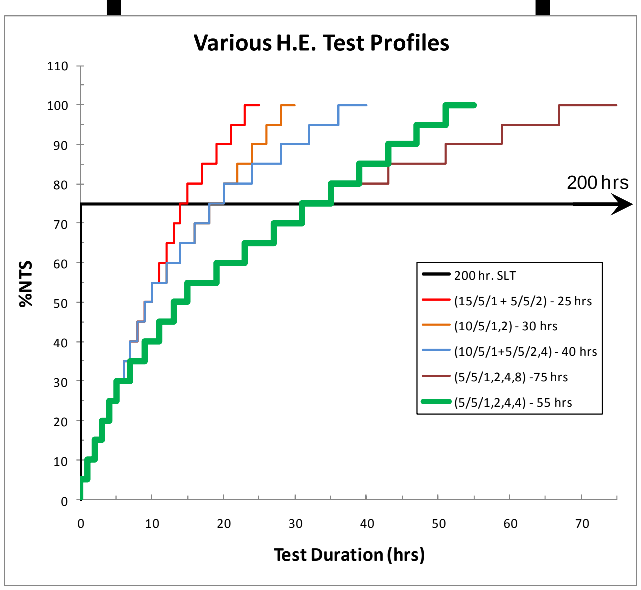
Specific Step Loads and Duration for Select ISL Tests of Interest

20-30 hr. ISL Tests Investigated

(50% + 10/5/2)			
20 hr test			
Step	% NTS	Time (hrs)	Cum Hrs
1	55	2	2
2	60	2	4
3	65	2	6
4	70	2	8
5	75	2	10
6	80	2	12
7	85	2	14
8	90	2	16
9	95	2	18
10	100	2	20
		20	

(15/5/1 + 5/5/2)			
25 hr test			
Step	% NTS	Time (hrs)	Cum Hrs
1	5	1	1
2	10	1	2
3	15	1	3
4	20	1	4
5	25	1	5
6	30	1	6
7	35	1	7
8	40	1	8
9	45	1	9
10	50	1	10
11	55	1	11
12	60	1	12
13	65	1	13
14	70	1	14
15	75	1	15
16	80	2	17
17	85	2	19
18	90	2	21
19	95	2	23
20	100	2	25
		25	

(10/5/1,2)			
30 hr test			
Step	% NTS	Time (hrs)	Cum Hrs
1	5	1	1
2	10	1	2
3	15	1	3
4	20	1	4
5	25	1	5
6	30	1	6
7	35	1	7
8	40	1	8
9	45	1	9
10	50	1	10
11	55	2	12
12	60	2	14
13	65	2	16
14	70	2	18
15	75	2	20
16	80	2	22
17	85	2	24
18	90	2	26
19	95	2	28
20	100	2	30
		30	

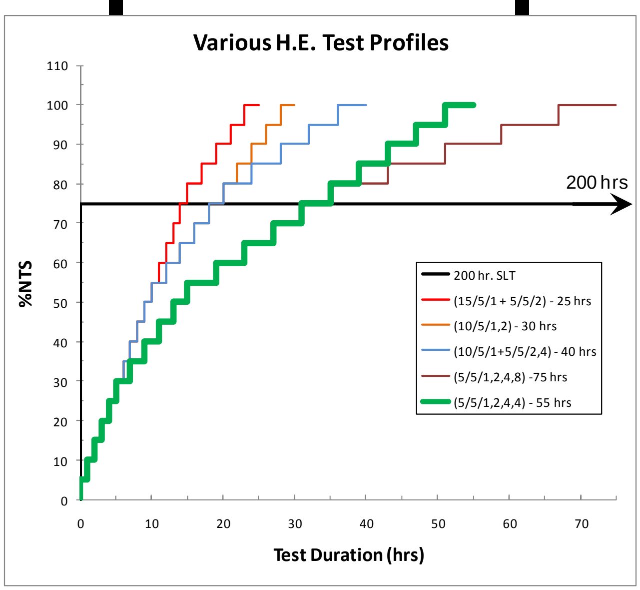


APPENDIX C, Con’t

Specific Step Loads and Duration for Select ISL Tests of Interest

Examples of 40 -75 hr. ISL Tests Investigated

(10/5/1 + 5/5/2,4)				(5/5/1,2,4,4)				(5/5/1,2,4,8)			
40 hr test				*55 hr test*				*75 hr test*			
Step	% FS	Time (hrs)	Cum Hrs	Step	% FS	Time (hrs)	Cum Hrs	Step	% FS	Time (hrs)	Cum Hrs
1	5	1	1	1	5	1	1	1	5	1	1
2	10	1	2	2	10	1	2	2	10	1	2
3	15	1	3	3	15	1	3	3	15	1	3
4	20	1	4	4	20	1	4	4	20	1	4
5	25	1	5	5	25	1	5	5	25	1	5
6	30	1	6	6	30	2	7	6	30	2	7
7	35	1	7	7	35	2	9	7	35	2	9
8	40	1	8	8	40	2	11	8	40	2	11
9	45	1	9	9	45	2	13	9	45	2	13
10	50	1	10	10	50	2	15	10	50	2	15
11	55	2	12	11	55	4	19	11	55	4	19
12	60	2	14	12	60	4	23	12	60	4	23
13	65	2	16	13	65	4	27	13	65	4	27
14	70	2	18	14	70	4	31	14	70	4	31
15	75	2	20	15	75	4	35	15	75	4	35
16	80	4	24	16	80	4	39	16	80	8	43
17	85	4	28	17	85	4	43	17	85	8	51
18	90	4	32	18	90	4	47	18	90	8	59
19	95	4	36	19	95	4	51	19	95	8	67
20	100	4	40	20	100	4	55	20	100	8	75
		40				**55**				**75**	



Approved for public release; distribution is unlimited.

APPENDIX D

Background ASTM E8-NTS Testing at Benét Laboratories

(all specimens from Lot HT)

Vendor Cert. Values

	NTS (ksi)	Fracture Load (lbs)
x-bar	**373**	**8978**
s_x	5.5	132

TEST DESCRIPTION	LOT	ID	NTS (ksi)	Fracture Load (lbs)
Notched Tensile Testing on As-Received Specimens	RSL HT	013	379	9115
		014	389	9365
		015	378	9092
		016	376	9048
		018	393	9460
		020	383	9209
		021	384	9239
		022	395	9502
		040	367	8829
		041	393	9449
		042	381	9158
		043	390	9376
		044	366	8795
		045	376	9047
		046	396	9523
		047	387	9318
		048	392	9420
		049	384	9245
		x-bar	**384**	**9233**
		s_x	9.0	217

TEST DESCRIPTION	LOT	ID	NTS (ksi)	Fracture Load (lbs)
Notched Tensile Testing after 375°F Stress Relief for 1 hr.	RSL HT	005	386.2	9290
		006	386.1	9286
		007	383.3	9220
		008	392.7	9444
		009	379.8	9136
		010	376.1	9045
		011	391.9	9427
		012	379.6	9130
		x-bar	**384**	**9247**
		s_x	5.9	143

TEST DESCRIPTION	LOT	ID	NTS (ksi)	Fracture Load (lbs)
Notched Tensile Testing after 225°F Bakeout for 8 hrs	RSL HT	001	380	8978
		002	369	8877
		003	387	9298
		004	397	9546
		x-bar	**383**	**9175**
		s_x	11.7	306

TEST DESCRIPTION	LOT	ID	NTS (ksi)	Fracture Load (lbs)
Notched Tensile Testing after Abrasive Blast w/o Mn-P	RSL HT	027	385.2	9265
		028	381.8	9184
		029	381.7	9181
		030	390.8	9400
		x-bar	**385**	**9257**
		s_x	4.3	103

TEST DESCRIPTION	LOT	ID	NTS (ksi)	Fracture Load (lbs)
Notched Tensile Testing after Abrasive Blast and Mn-P	RSL HT	031	391.1	9407
		032	397.2	9553
		033	393.1	9456
		034	395.7	9518
		x-bar	**394**	**9484**
		s_x	2.7	65

	NTS (ksi)	Fracture Load (lbs)
x-bar OF ALL SPECIMENS	**385**	**9259**
s_x OF ALL SPECIMENS	8.2	202

APPENDIX E

Statistical Analysis of Normal Dist. w/One-sided Tolerance Limit to Determine ISL-NFS$_P$ ($k_{99,95}$) for Bare, Uncoated, Notch Tensile Specimens

30 hr ISL profiile (10/5/1,2) ; ISL-NFS$_P$ ≥ 90% NTS

ID	Tester	Profile	Mat'l Lot	Fract. Load, lbs	Fract. Strength, ksi	NTS, lbs	NTS, ksi	% NTS	ISL-NFS$_P$ (lbs)	Comments	n	$k_{R,C}$
25	20A			8696.6	361.6	9258.7	385.1	93.9%	8342	Cracked upon loading to 95%	10	3.98
26	20B			8681.1	360.9	9258.7	385.1	93.8%		Cracked upon loading to 95%		
23	10A			8761	364.2	9258.7	385.1	94.6%	ISL-NFS$_P$ (% NTS)	Cracked upon loading to 95%		
24	10B			8602.1	357.6	9258.7	385.1	92.9%	90.1%	Cracked upon loading to 95%		
25	20A	(10/5/1,2)	RSL HT	8754.6	364.0	9258.7	385.1	94.6%		Cracked upon loading to 95%		
227	20B			8917.6	370.7	9258.7	385.1	96.3%	CV	Cracked upon loading to 100%		
226	20A			8708.1	362.0	9258.7	385.1	94.1%	1.2%	Cracked upon loading to 95%		
228	10A			8952.4	372.2	9258.7	385.1	96.7%		Cracked upon loading to 100%		
229	10B			8771	364.7	9258.7	385.1	94.7%		Cracked upon loading to 95%		
230	10A			8796.7	365.7	9258.7	385.1	95.0%		Cracked upon loading to 100%		
			x-bar =	8764.1	364.4			94.7%				
			s_x =	106.0	4.4			1.1%				

40 hr ISL profiile (10/5/1 + 5/5/2,4) ; ISL-NFS$_P$ ≥ 85% NTS

ID	Tester	Profile	Mat'l Lot	Fract. Load, lbs	Fract. Strength, ksi	NTS, lbs	NTS, ksi	% NTS	ISL-NFS$_P$ (lbs)	Comments	n	$k_{R,C}$
180	20A			8763.6	364.3	9258.7	385.1	94.7%	8097	Failed upon loading to 95%	10	3.98
182	20B			8642.6	359.3	9258.7	385.1	93.3%		Failed upon loading to 95%		
181	10B			8774.9	364.8	9258.7	385.1	94.8%	ISL-NFS$_P$ (% NTS)	Failed upon loading to 95%		
184	10A			8751.6	363.8	9258.7	385.1	94.5%	87.5%	Failed upon loading to 95%		
183	10B	(10/5/1 + 5/5/2,4)	RSL HTP	8338.1	346.7	9258.7	385.1	90.1%		Failed after 2.1 hrs at 90%		
186	20A			8913.4	370.6	9258.7	385.1	96.3%	CV	Faiiled upon loading to 100 %		
185	20B			8621.8	358.5	9258.7	385.1	93.1%	1.7%	Failed upon loading to 95%		
188	10A			8741.5	363.4	9258.7	385.1	94.4%		Failed upon loading to 95%		
207	10A			8659.6	360.0	9258.7	385.1	93.5%		Failed upon loading to 95%		
128	10B			8728.1	362.9	9258.7	385.1	94.3%		failed upon loading to 95%		
			x-bar =	8693.5	358.2			93.9%				
			s_x =	149.8	12.1			1.6%				

55 hr ISL profiile (5/5/1,2,4,4) ; ISL-NFS$_P$ ≥ 85% NTS

ID	Tester	Profile	Mat'l Lot	Fract. Load, lbs	Fract. Strength, ksi	NTS, lbs	NTS, ksi	% NTS	ISL-NFS$_P$ (lbs)	Comments	n	$k_{R,C}$
95	10B			8375.6	348.2	9258.7	385.1	90.5%	7972	Cracked after 2.9 hrs at 90%	10	3.98
93	10B			8804.1	366.0	9258.7	385.1	95.1%		Cracked upon loading to 95%		
92	20A			9009.9	374.2	9258.7	385.1	97.3%	ISL-NFS$_P$ (% NTS)	Cracked upon loading to 100%		
165	20B			8797	365.7	9258.7	385.1	95.0%	86.1%	Cracked after .5 hrs at 95%		
167	10A	(5/5/1,2,4,4)	RSL HTP	8636.8	359.1	9258.7	385.1	93.3%		Cracked upon loading to 95%		
166	20A			8719.8	362.5	9258.7	385.1	94.2%	CV	Cracked upon loading to 95%		
168	20B			8735.2	363.2	9258.7	385.1	94.3%	2.2%	Cracked upon loading to 95%		
169	10B			8582.5	356.8	9258.7	385.1	92.7%		Cracked upon loading to 95%		
171	20B			8691.7	361.4	9258.7	385.1	93.9%		Cracked upon loading to 95%		
170	10A			9027.3	375.3	9258.7	385.1	97.5%		Cracked upon loading to 100%		
			x-bar =	8738.0	363.2			94.4%				
			s_x =	192.6	7.9			2.1%				

75 hr ISL profiile (5/5/1,2,4,8) ; ISL-NFS$_P$ ≥ 80% NTS

ID	Tester	Profile	Mat'l Lot	Fract. Load, lbs	Fract. Strength, ksi	NTS, lbs	NTS, ksi	% NTS	ISL-NFS$_P$ (lbs)	Comments	n	$k_{R,C}$
100	20A			8433.3	350.6	9258.7	385.1	91.1%	7440	Cracked upon loading to 95%	10	3.98
99	10B			7890.2	328.0	9258.7	385.1	85.2%		Cracked soon after 85%		
97	10B			8372.9	348.1	9258.7	385.1	90.4%	ISL-NFS$_P$ (% NTS)	Cracked after 4.9 hrs at 90%		
98	10A			8782.0	365.1	9258.7	385.1	94.9%	80.4%	Cracked after 8 hrs at 90%		
96	20B	5/5/1,2,4,8	RSL HTP	8663.7	360.2	9258.7	385.1	93.6%		Cracking after 8 hrs at 90%		
178	20A			8329.5	346.3	9258.7	385.1	90.0%	CV	Cracking upon loading to 90%		
176	10A			8349.8	347.1	9258.7	385.1	90.2%	3.0%	Cracking upon loading to 90%		
177	10B			8729.1	362.9	9258.7	385.1	94.3%		Cracked upon loading to 95%		
175	20B			8509.3	353.8	9258.7	385.1	91.9%		Cracked upon loading to 95%		
179	10A			8575.9	356.5	9258.7	385.1	92.6%		Cracking upon loading to 95%		
10			x-bar =	8463.6	351.9			91.2%				
			s_x =	257.2	10.7			3.1%				

APPENDIX F

Results from 20-30 hr. ISL Testing

Test Date	ID	Machine	Supplier	Lot	Test	Condition	Pass/Fail	Test Duration (hrs)	% NTS	Lab
COATER A - 12/16/09										
12/16/2009	35	20A	RSL	HT/HTP	SLT	< 1 hr Delay	Fail	8	75	F&F
12/16/2009	37	10A	RSL	HT/HTP	SLT	< 1 hr Delay	Fail	5	75	F&F
12/16/2009	36	20B	RSL	HT/HTP	(10/5/1,2)	< 1 hr Delay	Pass	26	90	F&F
12/16/2009	38	10B	RSL	HT/HTP	(10/5/1,2)	< 1 hr Delay	Pass	26	90	F&F
COATER A - 1/10/10										
1/10/2010	39	20B	RSL	HT/HTP	SLT	< 1 hr Delay	Fail	8	75	F&F
1/10/2010	54	10B	RSL	HT/HTP	SLT	< 1 hr Delay	Pass	200	75	F&F
1/10/2010	50	10A	RSL	HT/HTP	(50% + 10/5/2)	< 1 hr Delay	Pass	16	90	F&F
1/10/2010	56	20A	RSL	HT/HTP	(50% + 10/5/2)	< 1 hr Delay	Pass	16	90	F&F
COATER A - 2/17/10										
2/17/2010	73	10B	RSL	HT/HTP	SLT	< 1 hr Delay	Fail	25	75	F&F
2/17/2010	75	20B	RSL	HT/HTP	SLT	< 1 hr Delay	Fail	2	75	F&F
2/17/2010	72	10A	RSL	HT/HTP	(15/5/1 + 5/5/2)	< 1 hr Delay	Fail	11	55	F&F
2/18/2010	79	N/A	RSL	HT/HTP	SLT	24 hr Delay	Fail	20	75	LRA
2/18/2010	76	N/A	RSL	HT/HTP	(15/5/1 + 5/5/2)	24 hr Delay	Pass	21	90	LRA
2/19/2010	78	N/A	RSL	HT/HTP	SLT	48 hr Delay	Pass	200	75	LRA
2/19/2010	77	N/A	RSL	HT/HTP	(15/5/1 + 5/5/2)	48 hr Delay	Pass	21	90	LRA
COATER A - 2/23/10										
2/23/2010	80	10A	RSL	HT/HTP	SLT	< 1 hr Delay	Fail	3	75	F&F
2/23/2010	82	20A	RSL	HT/HTP	SLT	< 1 hr Delay	Fail	10	75	F&F
2/23/2010	81	10B	RSL	HT/HTP	(10/5/1,2)	< 1 hr Delay	Fail	15	63	F&F
2/23/2010	83	20B	RSL	HT/HTP	(10/5/1,2)	< 1 hr Delay	Fail	17	70	F&F
2/25/2010	84	10A	RSL	HT/HTP	SLT	48 hr Delay	Fail	14	75	F&F
2/25/2010	85	10B	RSL	HT/HTP	(10/5/1,2)	48 hr Delay	Fail	18	69	F&F
2/26/2010	86	20A	RSL	HT/HTP	SLT	72 hr Delay	Pass	200	75	F&F
2/26/2010	87	20B	RSL	HT/HTP	(10/5/1,2)	72 hr Delay	Fail	25	80	F&F
COATER A - 3/9/10										
3/11/2010	94	20A	RSL	HT/HTP	SLT	48 hr Delay	Fail	19	75	F&F
3/11/2010	103	N/A	RSL	HT/HTP	SLT	48 hr Delay	Fail	7	75	LRA
3/11/2010	102	N/A	RSL	HT/HTP	(10/5/1,2)	48 hr Delay	Pass	200	90	LRA

The data in red indicate tests in which the ISL test passed but the SLT failed. Therefore, the conclusion is that these specific ISL tests are not as sensitive as the SLT test because they do not provide enough time for hydrogen to concentrate at the notch root and cause embrittlement.

One anomaly was observed on 2/26/2010 in which the SLT test passed after a 72 hr. RT delay but the ISL test failed.

APPENDIX G

Results from 40 hr. ISL Testing (10/5/1 + 5/5/2,4)

Test Date	ID	Machine	Supplier	Lot	Test	Condition	Pass/Fail	Test Duration (hrs)	% NTS	Lab
COATER A - 3/9/10										
3/9/2010	89	10B	RSL	HT	SLT	< 1 hr Delay	Pass	200	75	F&F
3/9/2010	91	20B	RSL	HT	SLT	< 1 hr Delay	Fail	6	75	F&F
3/9/2010	88	10A	RSL	HT	(10/5/1 + 5/5/2,4)	< 1 hr Delay	Fail	16	65	F&F
3/9/2010	90	20A	RSL	HT	(10/5/1 + 5/5/2,4)	< 1 hr Delay	Fail	10	49	F&F
3/10/2010	93	20B	RSL	HT	SLT	24 hr Delay	Fail	21	75	F&F
3/10/2010	92	20A	RSL	HT	(10/5/1 + 5/5/2,4)	24 hr Delay	Fail	13	58	F&F
3/11/2010	94	20A	RSL	HT	SLT	48 hr Delay	Fail	19	75	F&F
3/15/2010	103	N/A	RSL	HT	SLT	48 hr Delay	Fail	7	75	LRA
3/11/2010	95	20B	RSL	HT	(10/5/1 + 5/5/2,4)	48 hr Delay	Fail	24	80	F&F
3/12/2010	96	10A	RSL	HT	SLT	72 Hr Delay	Fail	6	75	F&F
3/12/2010	97	20A	RSL	HT	(10/5/1 + 5/5/2,4)	72 Hr Delay	Fail	19	75	F&F
COATER A - 3/17/10										
3/17/2010	105	20B	RSL	HT	SLT	< 1 hr Delay	Fail	2	75	F&F
3/17/2010	104	10A	RSL	HT	(10/5/1 + 5/5/2,4)	< 1 hr Delay	Fail	10	48	F&F
3/19/2010	109	20B	RSL	HT	SLT	48 hr delay	Pass	200	75	F&F
3/19/2010	108	10A	RSL	HT	(10/5/1 + 5/5/2,4)	48 hr delay	Pass	32	90	F&F
COATER A - 3/30/10										
3/30/2010	118	10A	RSL	HT	SLT	< 1 hr Delay	Fail	2	75	F&F
3/30/2010	119	20B	RSL	HT	(10/5/1 + 5/5/2,4)	< 1 hr Delay	Fail	9	44	F&F
3/31/2010	121	20B	RSL	HT	SLT	24 hr delay	Fail	5	75	F&F
3/31/2010	120	10A	RSL	HT	(10/5/1 + 5/5/2,4)	24 hr delay	Fail	12	55	F&F
4/1/2010	122	10A	RSL	HT	SLT	48 hr delay	Fail	8	75	F&F
4/1/2010	123	20A	RSL	HT	(10/5/1 + 5/5/2,4)	48 hr delay	Fail	24	80	F&F
4/2/2010	126	20B	RSL	HT	SLT	72 hr delay	Fail	24	75	F&F
4/2/2010	127	20A	RSL	HT	(10/5/1 + 5/5/2,4)	72 hr delay	Pass	30	90	F&F
4/5/2010	128-130	10A	RSL	HT	SLT	144 hr delay	Pass	200	75	F&F
4/5/2010	131-132	20A	RSL	HT	(10/5/1 + 5/5/2,4)	144 hr delay	Pass	31	88	F&F
COATER A - 4/12/10										
4/13/2010	136, 139	20A	RSL	HT	SLT	Furnace Bake	Pass	200	75	F&F
4/13/2010	134, 135, 138, 141	10B	RSL	HT	(10/5/1 + 5/5/2,4)	Furnace Bake	Pass	32	90	F&F
COATER A - 5/4/10										
5/5/2010	142	10A	RSL	HT	SLT	24 hr Delay	Fail	4	75	F&F
5/5/2010	143	10B	RSL	HT	(10/5/1 + 5/5/2,4)	24 hr Delay	Fail	11	49	F&F
5/8/2010	147	10B	RSL	HT	SLT	96 hr Delay	Fail	4	75	F&F
5/8/2010	148	20B	RSL	HT	(10/5/1 + 5/5/2,4)	96 hr Delay	Pass	28	85	F&F
5/10/2010	149	10A	RSL	HT	SLT	144 hr delay	Fail	12	75	F&F
5/10/2010	222	20B	RSL	HT	(10/5/1 + 5/5/2,4)	144 hr delay	Fail	13	57	F&F
COATER A - 5/14/10										
5/14/2010	016	10B	RSL	HTP	SLT	24 hr delay	Fail	4	75	F&F
5/14/2010	017	20A	RSL	HTP	(10/5/1 + 5/5/2,4)	24 hr delay	Fail	16	65	F&F
5/18/2010	019-022	10A	RSL	HTP	SLT	120 hr delay	Fail	14	75	F&F
5/18/2010	023-026	10B	RSL	HTP	(10/5/1 + 5/5/2,4)	120 hr delay	Pass	28	85	F&F
COATER B - 5/17/10										
5/20/2010	005	10B	RSL	HTP	SLT	72 hr delay	Fail	18	75	F&F
5/20/2010	007	20B	RSL	HTP	SLT	72 hr delay	Pass	200	75	F&F
5/20/2010	004	10A	RSL	HTP	(10/5/1 + 5/5/2,4)	72 hr delay	Fail	14	59	F&F
5/25/2010	008-011	10A	RSL	HTP	SLT	192 hr delay	Fail	5	75	F&F
5/25/2010	012-015	10B	RSL	HTP	(10/5/1 + 5/5/2,4)	192 hr delay	Fail	17	68	F&F

The data is orange is an anomaly in which the SLT test was not reproducible.

APPENDIX G, Con't

Results from 40 hr. ISL Testing (10/5/1 + 5/5/2,4)

Test Date	ID	Machine	Supplier	Lot	Test	Condition	Pass/Fail	Test Duration (hrs)	% NTS	Lab
COATER A -6/8/10										
6/9/2010	035-038	10A	RSL	HTP	SLT	Furnace Bake	Pass	200	75	F&F
6/9/2010	unk x 4	Mat. Eng.	RSL	HTP	SLT	Furnace Bake	Pass	200	75	Mat. Eng.
6/9/2010	039-042	20B	RSL	HTP	(10/5/1 + 5/5/2,4)	Furnace Bake	Pass	32	90	F&F
6/11/2010	065	10B	RSL	HTP	SLT	72 Hr Delay	Pass	200	75	F&F
6/11/2010	047	20A	RSL	HTP	(10/5/1 + 5/5/2,4)	72 Hr Delay	Pass	39	90	F&F
6/13/2010	048, 053, 060, 066	20A	RSL	HTP	SLT	120 hr delay	Pass	200	75	F&F
6/13/2010	unk x 4	Mat. Eng.	RSL	HTP	SLT	120 hr delay	Fail	7	75	Mat. Eng.
6/13/2010	056, 069, 070, 076	20B	RSL	HTP	(10/5/1 + 5/5/2,4)	120 hr delay	Fail	21	70	F&F
6/18/2010	unk x 4	Mat. Eng.	RSL	HTP	SLT	240 hr delay	Pass	200	75	Mat. Eng.
6/18/2010	061, 071, 074, 075	20B	RSL	HTP	SLT	240 hr delay	Fail	167	75	F&F
6/18/2010	050, 051, 055, 057	10A	RSL	HTP	(10/5/1 + 5/5/2,4)	240 hr delay	Pass	36	90	F&F
COATER A -7/13/10										
7/28/2010	H23	10A	Anachem	H23	SLT	336 hr delay	Pass	200	75	F&F
7/28/2010	H23	20B	Anachem	H23	(10/5/1 + 5/5/2,4)	336 hr delay	Fail	26	83	F&F
COATER A -7/28/10										
7/30/2010	H23	Mat. Eng.	Anachem	H23	SLT	48 hr delay	Fail	12	75	Mat. Eng.
7/30/2010	H23	20B	Anachem	H23	RSL 10/5/1 + 5/5/2,4	48 hr delay	Pass	32	90	F&F
8/2/2010	H23 x 4	Mat. Eng.	Anachem	H23	SLT	120 hr delay	Pass	90	75	Mat. Eng.
8/2/2010	H23 x 4	20B	Anachem	H23	(10/5/1 + 5/5/2,4)	120 hr delay	Pass	32	90	F&F
8/5/2010	H23	Mat. Eng.	Anachem	H23	SLT	192 hr delay	Pass	200	75	Mat. Eng.
8/5/2010	H23	20B	Anachem	H23	(10/5/1 + 5/5/2,4)	192 hr delay	Pass	32	90	F&F
COATER A - 8/12/10										
8/12/2010	GS-95	20B	Green Spec.	38	SLT	< 1 hr Delay	Fail	1	75	F&F
8/12/2010	GS-87	20A	Green Spec.	38	(10/5/1 + 5/5/2,4)	< 1 hr Delay	Pass	28	85	F&F
8/12/2010	083	10B	RSL	HTP	SLT	< 1 hr Delay	Fail	6	75	F&F
8/12/2010	089	10A	RSL	HTP	(10/5/1/ + 5/5/2,4)	< 1 hr Delay	Pass	31	88	F&F
8/13/2010	GS 35,43, 92,96	20B	Green Spec.	38	SLT	Furnace Bake	Pass	200	75	F&F
8/13/2010	GS 42, 90, 94, 98	10B	Green Spec.	38	(10/5/1 + 5/5/2,4)	Furnace Bake	Pass	32	90	F&F
8/17/2010	GS 33, 88, 91, 93	10A	Green Spec.	38	SLT	120 delay	Fail	54	75	F&F
8/17/2010	GS 41, 44, 85, 89	20A	Green Spec.	38	(10/5/1 + 5/5/2,4)	120 delay	Fail	24	80	F&F
8/30/2010	061, 063, 071, 085	20B	RSL	HTP	SLT	432 hr delay	Fail	72	75	F&F
8/30/2010	083, 086, 087, 164	10A	RSL	HTP	(10/5/1 + 5/5/2,4)	432 hr delay	Pass	32	90	F&F

The data in red indicate tests in which the ISL test passed but the SLT failed. What is disturbing in particular are the <1hr delay and 48 hr delay tests. The data in orange are anomalies in which the SLT test did not perform similar to another SLT test.

APPENDIX H

Results from 75 hr. ISL Testing (5/5/1,2,4,8)

Test Date	ID	Machine	Supplier	Lot	Test	Condition	Pass/Fail	Test Duration (hrs)	% NTS	Lab
COATER A - 2/2/10										
2/3/2010	68	10A	RSL	HT	SLT	Baked	Pass	200	75	F&F
2/3/2010	70	20A	RSL	HT	SLT	Baked	Pass	200	75	F&F
2/3/2010	69	10B	RSL	HT	(5/5/1,2,4,8)	Baked	Pass	59	90	F&F
2/3/2010	71	20B	RSL	HT	(5/5/1,2,4,8)	Baked	Pass	59	90	F&F
COATER A - 8/12/10										
8/30/2010	79, 63, 85, 161	20B	RSL	HTP	SLT	432 hr delay	Fail	72	75	F&F
8/30/2010	81, 88, 159, 162	10B	RSL	HTP	(5/5/1,2,4,8)	432 hr delay	Pass	43	80	F&F
COATER A - 9/2/10										
9/3/2010	H23 x 4	Mat. Eng.	Anachem	H23	SLT	Furnace Bake	Pass	200	75	Mat. Eng.
9/3/2010	H23 x 4	10B	Anachem	H23	(5/5/1/,2,4,8)	Furnace Bake	Pass	51	85	F&F
9/3/2010	H23	20B	Anachem	H23	(5/5/1/,2,4,8)	Furnace Bake	Pass	59	90	F&F
9/7/2010	H23 x 4	Mat. Eng.	Anachem	H23	SLT	120 hr delay	Fail	200	75	Mat. Eng.
9/7/2010	H23 x 4	20A	Anachem	H23	RSL 5/5/1,2,4,8	120 hr delay	Pass	59	90	Benet
COATER C - 9/9/10										
9/14/2010	4964	20B	Dirats	BG	SLT	120 hr delay	Pass	200	75	Benet
9/14/2010	4962	20A	Dirats	BG	(5/5/1/,2,4,8)	120 hr delay	Pass	58	90	Benet

The data in red indicate tests in which the ISL test passed but the SLT failed.

APPENDIX I

Results from 55 hr. ISL Testing (5/5/1,2,4,4)

Test Date	ID	Machine	Supplier	Lot	Test	Condition	Pass/Fail	Test Duration (hrs)	% NTS	Lab
COATER A - 1/27/10										
1/27/2010	61	10A	RSL	HT	SLT	< 1 hr Delay	Fail	6	75	F&F
1/27/2010	62	20A	RSL	HT	SLT	< 1 hr Delay	Fail	3	75	F&F
1/27/2010	59	10B	RSL	HT	(5/5/1,2,4,4)	< 1 hr Delay	Fail	16	53	F&F
1/27/2010	63	20B	RSL	HT	(5/5/1,2,4,4)	< 1 hr Delay	Fail	19	55	F&F
1/28/2010	67	Tens#2	RSL	HT	SLT	24 hr delay	Fail	13	75	LRA
1/28/2010	65	Tens#1	RSL	HT	(5/5/1,2,4,4)	24 hr delay	Fail	19	58	LRA
1/29/2010	64	Tens#2	RSL	HT	SLT	48 hr delay	Fail	7	75	LRA
1/29/2010	66	Tens#1	RSL	HT	(5/5/1,2,4,4)	48 hr delay	Fail	15	50	LRA
COATER A - 3/30/10										
4/1/2010	122	10A	RSL	HT	SLT	48 hr delay	Fail	8	75	F&F
4/1/2010	124	20B	RSL	HT	(5/5/1,2,4,4)	48 hr delay	Fail	27	65	F&F
4/2/2010	126	20B	RSL	HT	SLT	72 hr delay	Fail	24	75	F&F
4/2/2010	125	10A	RSL	HT	(5/5/1,2,4,4)	72 hr delay	Fail	39	80	F&F
4/5/2010	128, 129, 130	10A	RSL	HT	SLT	144 hr delay	Pass	200	75	F&F
4/5/2010	133	20B	RSL	HT	(5/5/1,2,4,4)	144 hr delay	Pass	43	85	F&F
COATER A - 5/4/10										
5/5/2010	142	10A	RSL	HT	SLT	24 hr Delay	Fail	4	75	F&F
5/5/2010	144	20A	RSL	HT	(5/5/1,2,4,4)	24 hr Delay	Fail	19	55	F&F
5/5/2010	145	20B	RSL	HT	(5/5/1,2,4,4)	24 hr Delay	Fail	12	42	F&F
5/8/2010	147	10B	RSL	HT	SLT	96 hr Delay	Fail	4	75	F&F
5/8/2010	146	10A	RSL	HT	(5/5/1,2,4,4)	96 hr Delay	Fail	23	60	F&F
5/10/2010	149	10A	RSL	HT	SLT	144 hr delay	Fail	12	75	F&F
5/10/2010	150	10B	RSL	HT	(5/5/1,2,4,4)	144 hr delay	Fail	18	54	F&F
COATER A - 5/13/10										
5/14/2010	016	10B	RSL	HTP	SLT	24 hr delay	Fail	4	75	F&F
5/14/2010	018	20B	RSL	HTP	(5/5/1,2,4,4)	24 hr delay	Fail	39	80	F&F
5/18/2010	019, 020, 021, 022	10A	RSL	HTP	SLT	120 hr delay	Fail	14	75	F&F
5/18/2010	027, 028, 029, 030	20B	RSL	HTP	(5/5/1,2,4,4)	120 hr delay	Fail	39	80	F&F
COATER B - 5/17/10										
5/20/2010	005	10B	RSL	HTP	SLT	72 hr delay	Fail	18	75	F&F
5/20/2010	007	20B	RSL	HTP	SLT	72 hr delay	Pass	200	75	F&F
5/20/2010	006	20A	RSL	HTP	(5/5/1,2,4,4)	72 hr delay	Pass*	43	85	F&F

*The data in orange is one ISL test which passed based on the NFS results. However, SEM fractography identified a region of HE (IG cracking). Note that one head-to-head SLT test passed while another failed. This is discussed further in the report.

APPENDIX I, Con't

Results from 55 hr. ISL Testing (5/5/1,2,4,4)

Test Date	ID	Machine	Supplier	Lot	Test	Condition	Pass/Fail	Test Duration (hrs)	% NTS	Lab
COATER A -9/22/10										
1/4/2011	103	20A	RSL	HTP	SLT	3 mo delay	Pass	206	75	F&F
1/4/2011	114	10A	RSL	HTP	(5/5/1,2,4,4)	3 mo delay	Pass	51	95	F&F
COATER D -12/15/10										
12/19/2010	195	10B	RSL	HTP	SLT	96 hr delay	Pass	200	95	F&F
12/19/2010	194	20A	RSL	HTP	(5/5/1,2,4,4)	96 hr delay	Pass	50	90	F&F
COATER A -2/9/11										
2/10/2011	H30 x 4	Mat. Eng.	Anachem	H30	SLT	Furnace Bake	Pass	200	75	Mat. Eng.
2/10/2011	H30	10A	Anachem	H30	(5/5/1,2,4,4)	Furnace Bake	Pass	51	95	F&F
2/10/2011	H30	10B	Anachem	H30	(5/5/1,2,4,4)	Furnace Bake	Pass	51	95	F&F
2/14/2011	H30 x 4	Mat. Eng.	Anachem	H30	SLT	120 hr Delay	Pass	200	75	Mat. Eng.
2/14/2011	H30	10A	Anachem	H30	(5/5/1,2,4,4)	120 hr Delay	Fail	31	70	F&F
2/14/2011	H30	10B	Anachem	H30	(5/5/1,2,4,4)	120 hr Delay	Pass	51	95	F&F
COATER A-3/09/11										
3/10/2011	H30 x 4	Mat Eng	Anachem	H30	SLT	Furnace Bake	Pass	200	75	Mat. Eng.
3/10/2011	H30	10A	Anachem	H30	(5/5/1,2,4,4)	Furnace Bake	Pass	48	91	F&F
3/10/2011	H30	10B	Anachem	H30	(5/5/1,2,4,4)	Furnace Bake	Pass	51	95	F&F

APPENDIX J

Low Risk of HE in Armament Components from Mn-P

Mn-P of Armament Components & H.E.

Why haven't armament components been embrittled by H?

Why haven't armament components been embrittled by H from Mn-P?

- Components are typically <HRC 51-53
- Cyclic tensile stresses (at high strain rates) vs. sustained load
- Abrasive blasting may impart surface compressive residual stresses that may retard H.E.
- Nitrided/carburized/case hardened components have surface compressive residual stresses
- Long time for H egress between Mn-P and service operation
- Potential for more effective hydrogen trapping in higher V gun steels (G.L. Spencer)

TECHNOLOGY DRIVEN. WARFIGHTER FOCUSED.

7

G.N. Vigilante, Benet Quarterly Management Review, 10 Feb. 2010.

More info on G.L. Spencer work can be found in Ref. [2].